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ESSENTIALS FOR THE CPI PROFESSIONAL

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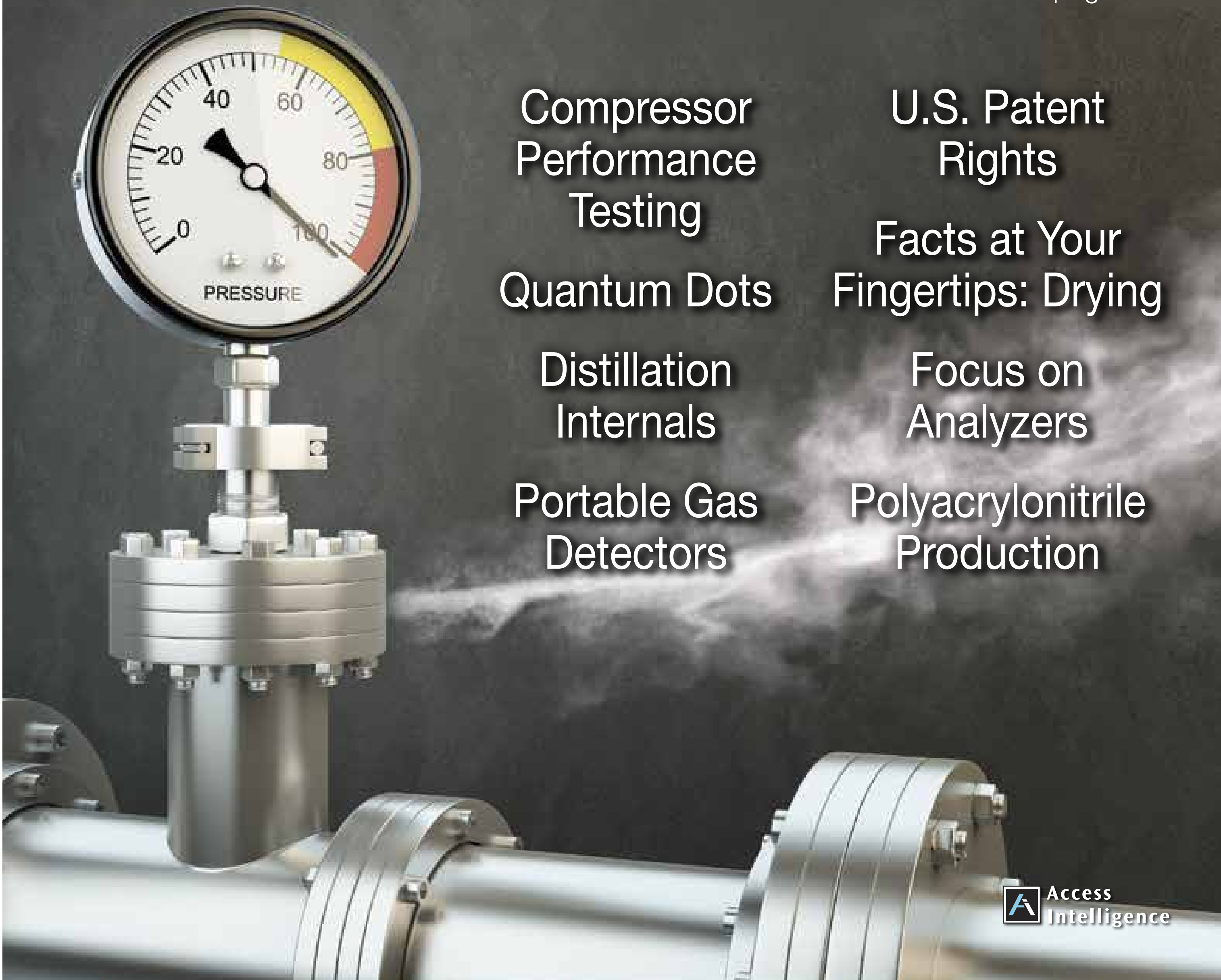

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BPA

Editor's Page

The far reach of the CPI

To determine the impact of the chemical industry's contributions to the global economy, the International Council of Chemical Associations (ICCA; www.icca-chem.org) commissioned Oxford Economics to provide a detailed assessment. Last month, the results were released in the report: *The Global Chemical Industry: Catalyzing Growth and Addressing Our World's Sustainability Challenges* (www.icca-chem.org/economicanalysis).

Some of the major findings in the report are: 1) the chemical industry added $1.1 trillion to the world's gross domestic product (GDP) and employed 15 million people; 2) an additional $4.20 was generated elsewhere in the global economy for every $1 generated by the chemical industry; 3) companies in the chemical industry spent an estimated $3 trillion with their suppliers in 2017; and 4) the chemical industry invested approximately $51 billion in research and development. As defined in this study, the chemical industry did not include refined petroleum products or pharmaceutical products.

Much more than chemicals

Looking beyond the economic impact, the products and services provided by the chemical process industries (CPI) are extraordinarily broad and touch most aspects of our daily lives. In fact, according to the report, the ICCA estimates that over 95% of all manufactured goods rely on some type of industrial chemical process.

Consider advances being made in materials of construction: Lighter weight components for automobiles and airplanes; improved insulation and coatings for use in buildings; chemistry to improve concrete; and materials that make new biomedical devices possible. Many advances in electronics are enabled by CPI products, such as battery technologies, organic light emitting diodes and the latest developments in quantum dots (see the Newsfront on pp. 14–17). Fertilizers and other agrochemicals help to increase food supplies; produced fibers provide clothing and protection, such as in bullet-proof vests; and the variety and uses of plastics are too numerous to list. Contributions to solar panels and energy storage are helping to shape the transformations in the energy sector. Advances in water treatment help to provide an essential clean water supply. And contributions from the CPI toward a sustainable future take many forms, including bio-based processes, recycling developments and technologies related to carbon capture.

A source of innovation

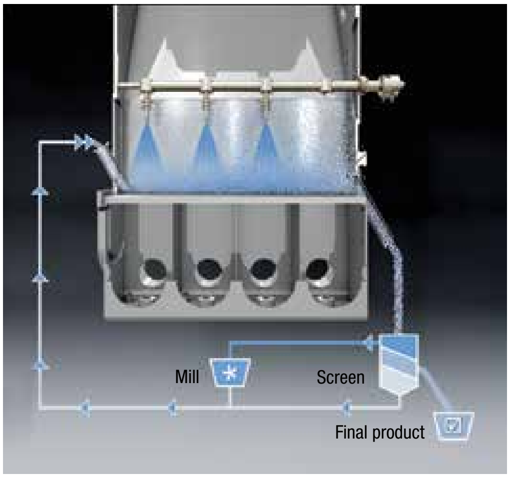
As mentioned in the report, the chemical industry invests heavily in research and development. Chinese chemical companies were found to have spent the most in 2017 at $15 billion, followed by U.S. companies at $12 billion. A look through our Chementator Dept. (pp. 7–11) gives a glimpse into some of the new technologies under development.

Patents are a good indication of innovation. In the U.S., the chemical sector is one of the most patent-intensive alongside electronics, according to the report (for more on patents, see the article on U.S. Patent Rights on pp. 49–56).

The principles of chemical engineering offer a sound basis to work in a wide variety of areas. This versatility is in large part what attracted me to the field and set me on my own journey as a chemical engineer. The advances that are being made and are contributing to so many areas keep the chemical sector one of the most exciting to work in, as well as one of the most influential across other fields. ■

Dorothy Lozowski, Editorial Director

This P-recovery process is market-ready



Glatt

At PowTech 2019 this month (Nuremberg, Germany), Glatt Ingenieurtechnik GmbH (Weimar, Germany; www.glatt.com) introduced a waste-free process that recycles the phosphorus from sewage-sludge ash to produce ready-to-use, high-quality fertilizers. The market-ready process fulfills the legal obligation of the German fertilizer ordinance (DüMV), which requires waste-disposal companies to recover phosphorus, and can recover virtually all of the phosphorus from the ash of incinerated sewage sludge.

In the first step of the two-stage PHOS4green process (diagram), a suspension is produced from the phosphate-containing ash, a mineral acid (for example, phosphoric acid) and, depending on the objective, other components. This acid treatment macerates the ash, and converts insoluble phosphates into a form that can be absorbed by plants. In the second step, the suspension is sprayed into a fluidized-bed granulator, which is blown with hot air. As the water evaporates, the granules grow in size before being discharged. Product is screened to separate granules of the desired size from over- and undersized solids, which are recirculated to the granulator.

"The process recycles 100% of the ash," says Jan Kirchhof, senior sales manager Process and Plant Engineering at Glatt. "By flexibly adapting the recipes, a wide variety of fertilizers, including complex varieties (NP, PK, NPK), can be produced, which can be placed on the market as new products," he says.

The process has been tested on various sludge ashes, from mono-incineration plants, in a pilot plant at Glatt's Technology Center in Weimar. The pilot unit can produce approximately 60 kg/h of standard fertilizer. Several engineering phases have already been completed for the first large-scale commercial plant, which is now in the approval phase. Groundbreaking for this project is anticipated in the next few weeks. The modular system can be configured for the desired capacity, with various configurations handling a capacity of 10,000–40,000 ton/yr of sludge ash. Nevertheless, smaller and larger plant set-ups are possible.

A new membrane for forward osmosis

Many desalination technologies have been developed, but they usually require a large amount of energy. Forward osmosis (FO) has the potential to become an excellent alternative desalination technology due to its lower energy consumption. Nano structured materials have also attracted attention, particularly metal organic frameworks (MOFs), due to their exceptional separation properties, controllable pore sizes, adsorption affinities and high porosity. To use an MOF as an effective membrane, an MOF is deposited onto an inorganic support to provide mechanical strength and chemical resistance. Increased attention has been given to a subclass of MOFs: the zeolitic imidazolate frameworks (ZIFs) that consist of zinc or cobalt ions coordinated by an imidazole-based linker. Under this group, ZIF-8 has been the most commonly studied for various separation applications.

Now a team from Universiti Teknologi Malaysia (Skudai, Malaysia; www.utm.my) has carried out the in-situ synthesis of ZIF-8 membranes using the solvothermal process onto alumina hollow fiber in the presence of ZnO, NiO and polydopamine (PDA). In addition, the different molar ratio of sodium formate in synthesis solution was studied on the membrane characterization and performance. The team evaluated the performance of ZIF-8 membranes in desalination, using FO. "To the best of our knowledge, no studies have reported on ZIF-8 membranes for desalination using forward osmosis," says the team.

The team found ZIF-8 to be a promising membrane material for desalination using FO. However, it said, improvements in membrane morphology to prepare a well-intergrown, continuous and thin membrane are needed to enhance its performance in terms of water flux, reverse-solute flux and salt rejection.

Edited by:
Gerald Ondrey

'GREEN' STEELMAKING

Seamlessly following the successful completion of the GrInHy project (Green Industrial Hydrogen via reversible high-temperature electrolysis) in February, the GrInHy2.0 project was launched last month at the Salzgitter Flachstahl GmbH steelworks (Germany; www.salzgitter-flachstahl.de). Together with partners Sunfire GmbH (Dresden, Germany; sunfire.de), Paul Wurth S.A., Tenova S.p.A., French research center CEA and Salzgitter Mannesmann Forschung GmbH, the world's most powerful high-temperature electrolyzer (HTE) is being constructed for the energy-efficient production of H_2. The GrInHy2.0 project has an overall budget of € 5.5 million.

GrInHy2.0 marks the first implementation of an HTE with a power rating of 720 kW in an industrial environment. By the end of 2022 it is expected to have been in operation for at least 13,000 h, producing a total of around 100 metric tons of high-purity (99.98 %) H_2, which will be used for annealing processes in the integrated steelworks as a replacement for H_2 produced from natural gas.

H_2 as a reducing agent is also a central element in SALCOS (Salzgitter Low-CO_2 Steelmaking), the Salzgitter Group's concept for reduced-CO_2 steel production, is set to replace the carbon previously required to reduce iron ore to metallic iron. SALCOS is based on elements of various proven technologies, allowing the concept to be implemented swiftly at an industrial level.

By increasing the scale of the HTE, the GrInHy2.0 project will have the ability to extensively trial and test the integration of "green" H_2 into the steelworks processes. To do so, the gaseous product of the

(Continues on p. 8)

Sunfire-HyLink electrolyzer will first be compressed and dried in the H_2-processing unit supplied by Paul Wurth. Salzgitter Flachstahl will be responsible for operating the plant and feeding the gas into the company's own H_2 network.

In parallel with this, CEA will conduct multi-year trials of the electrolyzer stacks that form the central elements of HTE technology. Tenova will support the project through a technical and economic study to accompany the decarburization of European steel industry through the conversion at a very low CO_2, green-H_2-based steel production. Salzgitter Mannesmann Forschung is responsible for project coordination and management.

TiO_2 COATING

An antimicrobial titanium dioxide coating, claimed to be much more effective than previous titanium dioxide coatings, has been developed by a team led by professor Susan Krumdieck of the University of Canterbury (Christchurch, New Zealand; www.canterbury.ac.nz), and researchers from the University of Grenoble Alpes (Grenoble, France; www.univ-grenoble-alpes.fr).

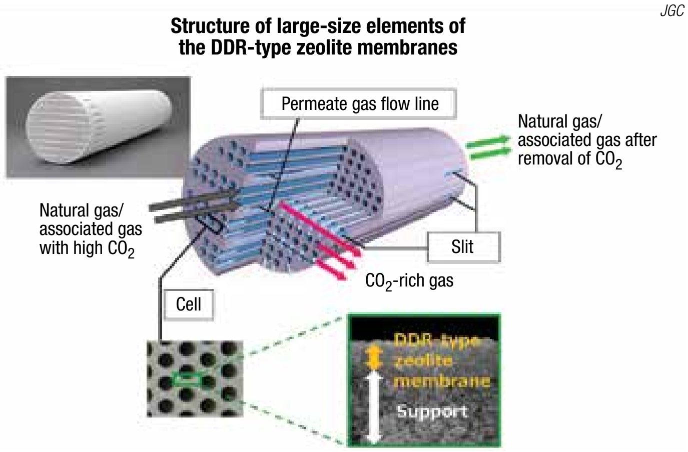
TiO_2 has received much attention as a possible antimicrobial material due to its

(Continues on p. 9)

Zeolite membranes to undergo large-scale testing for CO_2 capture

In late February, construction began on a test facility that will be used to demonstrate a CO_2-capture process being developed by JGC Corp. (Yokohama, Japan; www.jgc.com) and Japan Oil, Gas and Metals National Corp. (JOGMEC; Tokyo; www.jogmec.go.jp). When the Texas-based facility is completed, it will operate for one year to demonstrate a zeolite-type membrane for removing CO_2 from associated gas that is generated during petroleum production. The site produces 3 million ft^3/d of associated gas.

JGC



For the demonstration, the facility will use DDR-type zeolite membranes, which have been under development since 2008 by JGC and the ceramics producer NKG Insulators, Ltd. (Tokyo; www.ngk-insulators.com). The test will confirm the performance of the process using large-size elements (180 mm dia.; 1,000 mm length) of the DDR-type zeolite membrane. The separation process (diagram) has been jointly developed by NGK and JGC since 2008. The DDR-type zeolite membranes will be manufactured by NGK using its advanced membrane-production technology. The membranes' "outstanding" CO_2 selectivity and high-pressure resistance have been verified in tests carried out at the company's research facility.

JGC expects the process can be applied for both CO_2 recovery from associated gas during oil production, and CO_2 removal during natural gas processing. Current methods generally use polymer-membrane separation processes for the former, and chemical absorption processes for the latter. However, these methods both have drawbacks. For example, significant deterioration of polymer membranes often occurs when the associated gas contains high concentrations of CO_2, resulting in costly membrane replacement. In contrast, the DDR-type zeolite membrane is highly durable, even under high CO_2 concentration conditions.

High CO_2 concentrations in natural-gas processing can also lead to increased costs associated with chemical absorption methods, due to increased solvent usage and energy needed for solvent regeneration. For this application, the zeolite-based membrane could be used before the absorption step to significantly reduce these costs.

A new catalyst for making renewable feedstocks

The compound 2,5-furandicarboxylic acid (FDCA) is an attractive raw material that can be used to create polyethylene furanoate, which is a bio-polyester with many applications. One way of making FDCA is through the oxidation of 5-hydroxymethyl furfural (HMF), a compound that can be synthesized from cellulose. However, the necessary oxidation reactions require the presence of a catalyst, and many of the catalysts studied thus far involve precious metals. It is known that manganese oxides combined with certain metals (such as iron and copper) can be used as catalysts. Now, a research team from the Tokyo Institute of Technology (Yokohama Campus, Japan; www.msl.titech.ac.jp/~hara/index-e.html) has reported that manganese dioxide (MnO_2) can be used by itself as an effective catalyst if the crystals made with it have the appropriate structure.

The team, led by associate professor Keigo Kamata and professor Michikazu Hara, worked to determine which MnO_2 crystal structure would have the best catalytic activity for making FDCA, and why. They inferred through computational analyses and the available theory that the structure of the crystals was crucial because of the steps involved in the oxidation of HMF. To verify this, they synthesized various types of MnO_2 crystals, and then compared their performance through numerous analyses. Of these crystals, β-MnO_2 was the most promising because of its active planar oxygen sites. Not only was its vacancy formation energy lower than that of other structures, but the material itself was proven to be very stable, even after being used for oxidation reactions on HMF.

The team proposed a new synthesis method to yield highly pure β-MnO_2 with a large surface area in order to improve the FDCA yield and accelerate the oxidation process even further. "The synthesis of high-surface-area β-MnO_2 is a promising strategy for the highly efficient oxidation of HMF with MnO_2 catalysts," says Kamata.

Nanostructured catalyst converts CO_2 to ethanol electrochemically

Reducing carbon dioxide to useful fuel molecules is a highly desired objective, but is difficult because the required reactions are energetically unfavorable, and a cost-effective and robust catalyst for the reduction has been elusive. Now researchers at Oak Ridge National Laboratory (ORNL; Oak Ridge, Tenn.; www.ornl.gov) may have found such a catalyst — nanostructured carbon with embedded copper nanoparticles — that can convert CO_2 to ethanol in an electrochemical cell.

"The reaction between CO_2 and the catalyst is kind of a reverse combustion process that takes place in a modified fuel cell," explains Adam Rondinone, ORNL senior scientist. "It offers a pathway for using renewable electricity to make carbon fuels from a greenhouse gas."

In March, the ORNL catalyst and electrochemical cell technology was licensed by ReactWell LLC (New Orleans, La.; www.reactwell.com) for further development, and inclusion in industrial process units. "The catalyst is a heavily textured graphene with copper nanoparticles lodged into the surface," says Rondinone. "The graphene forms carbon 'nanospikes' that orient the CO_2 molecules to influence the reaction mechanism." The catalyst material forms localized electric fields, which focus the electrochemical reactivity so that the reaction pathway forms the desired products.

At the cathode of the cell, the catalyst reduces CO_2 to CO on the catalyst surface. CO is a reaction intermediate that dimerizes and combines with hydrogen from water (which is co-reduced in the cell) to form ethanol. Oxygen is evolved at the anode.

ReactWell CEO Brandan Iglesias says the first application for the licensed ORNL technology is making ethanol for alcoholic spirits, because it is a low-volume, high-margin market, he explains, and is attractive to enter with this novel electric alcohol technology. Eventually, the technology would be used at petroleum refineries, where CO_2 from exhaust gas could be captured and converted into ethanol to upgrade transportation fuel stocks.

photocatalytic activity (PCA) and self-cleaning properties. It has selective spectral absorption in the ultraviolet range, but is transparent to visible light. When titanium is exposed to ultraviolet (UV) light, it breaks down water vapor in air to produce free oxygen radicals, which attack organic matter, such as the membranes of bacterial cells.

Krumdieck and her colleagues grow TiO_2 coatings directly on stainless steel using a pulsed chemical-vapor deposition (CVD) technique she developed. An ultrasonic atomizer is used to make a fine mist of a TiO_2 precursor, which is pulse-sprayed into the CVD chamber. This technique increases the rate at which the TiO_2 crystals grow, resulting in a 10-µm-thick coating with an interesting structure: vertical columns of stacked 15–20-nm-thick single crys-

(Continues on p. 10)

tals of TiO_2 are interspersed with pinecone-like polycrystalline structures. The coating has a surface area 100 times larger than previous TiO_2 coatings made with nanoparticles, resulting in more bacteria-killing oxygen. There is carbon on the crystal surfaces in spaces between the nanostructures, which helps absorb visible-light photons, and feeds them to the TiO_2, Krumdieck says.

The team tested the coating by spreading *E.coli* culture on a sample and exposing it to UV light and white light for four hours. UV exposure killed all of the bacteria every time, while visible light killed over 99% of the bacteria.

WASTEWATER TREATMENT

A new pilot plant to treat industrial wastewater is being built that could potentially reduce the amount of liquid waste by over 90%. Located at a semiconductor manufacturer in Singapore, the new plant will also recover precious metals from the treated water. The plant is being built jointly by the Separation Technologies Applied Research and Translation (START) Center — a national-level facility to develop and commercialize innovative separation and filtration technologies — and Memsift Innovations Pte Ltd. (www.memsift.com), a local water technology firm specializing in zero-liquid discharge water-treatment systems.

The pilot plant will use a water-treatment system that leverages a new type of hollow-fiber membrane invented by professor Neal Chung at the National University of Singapore (NUS; http://nus.edu.sg). Unlike conventional hollow-fiber membranes, which resemble spaghetti noodles with a hollow core, the new tri-bore hollow-fiber membranes have three hollow cores, allowing for a water flowrate that is about 30% higher.

Under a new research partnership and licensing agreement, the START Center and Memsift Innovations will jointly build the wastewater-treatment plant with the tri-bore hollow-fiber membranes, which can treat up to 5,000 L/d. This pilot plant is expected to help the company save up to 1.6 million L/yr of water, resulting in a savings of $250,000 in disposal cost.

The new pilot plant is expected to be commissioned in the second quarter of 2019, and the piloting results used for commercializing the technology.

SMART SOLVENT EXTRACTION

At the SME Annual Conference and Expo (February 24–27; Denver, Colo.), Solvay S.A. (Brussels, Belgium; www.solvay.com) and Codelco (Santiago, Chile; www.codelco.com), the world's largest copper producer, launched SolvExtract — a first-

(Continues on p. 11)

Mechanochemistry performs cross-coupling reactions

Palladium-catalyzed cross-coupling reactions are one of the most powerful and versatile methods to synthesize a wide range of complex functionalized molecules. However, the development of solid-state cross-coupling reactions remains extremely limited. Now, Hajime Itoh, Koji Kubota and colleagues at Hokkaido University (Sapporo, Japan; https://labs.eng.hokudai.ac.jp/labo/organoelement) reported a rational strategy that provides a general entry to palladium-catalyzed Buchwald-Hartwig cross-coupling reactions in the solid state. The key finding of this study is that olefin additives can act as efficient molecular dispersants for the palladium-based catalyst in solid-state media to facilitate solid-state cross-coupling. Their strategy could inspire the development of industrially attractive, solvent-free palladium-catalyzed cross-coupling processes for other valuable synthetic targets.

Conventionally, palladium-catalyzed cross-coupling reactions of liquid and solid substrates are conducted in organic solvents. Researchers sought to re-design palladium-based catalyst systems for the solid state, which could potentially unlock versatile applications for solid-state synthesis.

They have developed a rational strategy for a potentially general and scalable solid-state palladium-catalyzed cross-coupling reaction using mechanochemistry. Whereas the palladium-catalyzed cross-coupling of neat liquids proceeds readily in ball mills, similar reactions using solid reactants remain challenging. However, they discovered that the addition of small amounts of olefins dramatically accelerates the C–N cross-coupling of such solid substrates. The examination of palladium nanoparticles, which were obtained from these reaction mixtures, by transmission electron microscopy (TEM) suggested that the olefin additives can act as efficient molecular dispersants for the palladium catalysts in solid-state media and thus facilitate this challenging solid-state cross-coupling. They expect that the strategy developed in this study could unlock broad areas of chemical space for palladium-catalyzed solid-state syntheses of valuable synthetic targets in various scientific fields.

CO_2 scrubber supplies raw material for oxalic acid production

A novel carbon-capture project at Michigan Technical University (MTU; Houghton, Mich.; www.mtu.edu) couples the collection of carbon dioxide from power-plant fluegas with a system to use the captured CO_2 as a raw material to make oxalic acid, which can be used in the mining industry to leach rare earth elements from ore.

"Coal-fired power plants can be acceptable economically and environmentally if you can use the CO_2 for a productive purpose, rather than allowing it into the atmosphere, where it is harmful, or sequestering it underground, where it is wasted," says MTU professor and project leader Komar Kawatra.

In the first phase of the project, a sodium carbonate solution is pumped to the top of a scrubbing column, where CO_2-rich exhaust gas from the MTU power plant is bubbled through the solution. "The scrubber is able to reduce the CO_2 content in the exhaust gas from 8% to less than 1%," says MTU researcher and doctoral student Sriram Valluri.

Waste heat from the power plant is used to regenerate CO_2 from the sodium carbonate after it is captured. The sodium carbonate scrubber is less expensive than conventional carbon-capture systems based on amines, and the chemicals are much less toxic than amines, the team says.

In the second stage of the project, the CO_2 that has been captured from the exhaust gas is reacted in an electrolytic cell to convert it to the two-carbon dicarboxylic acid known as oxalic acid. Locally produced oxalic acid could boost the production of rare-earth elements in the U.S., says Kawatra.

Currently, the CO_2 scrubber is operating at pilot scale on the MTU power plant, while the oxalic acid production system is operating at bench scale, doctoral student Victor Claremboux says. The next step in the project is to scale up the oxalic acid system.

of-its-kind application-based digital platform for mining operations. SolvExtract enables users to make more informed decisions faster, thus reducing process variability and boosting productivity.

SolvExtract is the first of Solvay's new digital solutions for mining operations, and is said to be the only digital platform today dedicated to reagent optimization. The platform enables data flows between a mining operation and Solvay's proprietary solvent extraction (SX) modeling software. Using this software, Solvay personnel are able to monitor key variables of the SX plant, such as Cu recovery and Cu transfer, comparing actual values and trends to model predictions, to identify and analyze deviations. Leveraging a dashboard, custom alerts and expert analysis, Solvay quickly notifies plant operators when issues arise and offers recommendations through the platform. ❑

First applications announced for a supramolecular chemistry technology

A novel and versatile performance chemistry that enables a variety of supramolecular assemblies has been released for its first commercial applications. The molecules, tradenamed AqBit, are characterized by their ringed structures, barrel-like shapes and ability to capture, hold and release other materials. The company Aqdot Ltd. (Cambridge, U.K.; www.aqdot.com) is commercializing AqBit for a host of applications, all involving supramolecular assemblies held together by non-covalent interactions, such as hydrogen bonds and hydrophobic interactions.

The first commercial application for the technology is in air quality, and is a system designed to capture molecules such as thiols or amines, main components of household and industrial odors. "AqBit captures the odor-causing species very effectively, rather than just masking them with other fragrances," says Jing Zhang, co-founder of Aqdot.

A second commercial application is in the personal care industry, where there has been a demand for more naturally derived ingredients. The natural components in personal care products are desirable environmentally and from a consumer preference angle, says Zhang, but they have difficulty forming the stable oil-in-water emulsions needed for formulating these products. The AqBit-enabled naturally derived emulsifier AqStar M1 stabilizes the emulsions to allow performance with the desired ingredients while delivering formulation flexibility and distinctive aesthetics, she adds.

In other instances, AqBit acts as a supramolecular "linker" for polymers to form a capsule around some target species, such as enzymes, agrochemicals and pharmaceuticals. In life sciences applications, AqBit-enabled capsules can be used to deliver genes for therapy. Then, using a chemical trigger, such as a pH change, or a redox reaction, the capsules can be induced to release their payloads.

The commercial applications have been enabled by the company's development of a proprietary, large-scale manufacturing process. The company plans to develop a host of products based on the supramolecular chemistry technology. ■

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Plant Watch

Asahi Kasei plans LIB separator capacity expansions

March 14, 2019 — Asahi Kasei Corp. (Tokyo, Japan; www.asahi-kasei.co.jp) will enhance its manufacturing infrastructure for lithium-ion battery (LIB) separator products at the company's existing facilities in Moriyama, Japan and North Carolina, with completion scheduled for 2021. The investment for this project is around ¥30 billion ($270 million). This expansion will result in a 300 million-m^2/yr rise in wet-process capacity and a 150 million-m^2/yr rise in dry-process capacity.

Outotec to construct sulfuric acid plant in Morocco

March 13, 2019 — Outotec Oyj (Espoo, Finland; www.outotec.com) signed a contract with OCP Group in Morocco for the construction of a sulfuric acid plant for fertilizer production. The €80-million order includes advanced proprietary technologies, including a heat-recovery system, as well as a converter, absorption towers and an acid-distribution system.

Invista signs first Middle East technology license for purified terephthalic acid

March 12, 2019 — Invista (Wichita, Kan.; www.invista.com) and Pan-Asia PET Resin (Guangzhou) Co. reached an agreement for the licensing of Invista's purified terephthalic acid (PTA) process technology at a new plant to be located in Jizan Economic City, Saudi Arabia. The planned PTA production capacity is 1.25 million metric tons per year (m.t./yr). This project signifies Invista's first technology license in the Middle East.

Celanese to increase production capacity for thermoplastic co-polyester in Italy

March 6, 2019 — Celanese Corp. (Dallas, Tex.; www.celanese.com) plans to expand its thermoplastic co-polyester production unit at the Donegani facility in Ferrara, Italy. Celanese recently added a solid-state polymerization unit at the Donegani facility, which started up in September 2018. Celanese expects to further expand the unit's capacity by adding another polymerization line to be completed in the next 15 to 18 months.

Mitsui to construct plant for long glass-fiber-reinforced polypropylene in China

March 1, 2019 — Mitsui Chemicals, Inc. (MCI; Tokyo; www.mitsuichem.com) will set up a new facility to produce 3,500 m.t./yr of long glass-fiber-reinforced polypropylene (LGFPP) at Chinese manufacturing subsidiary Mitsui Advanced Composites (Zhongshan) Co., which will join existing LGFPP sites in Japan and the U.S. When the new facility commences operations in September 2020, MCI's total production capacity for LGFPP will increase to 10,500 m.t./yr.

ExxonMobil moves forward with construction of Baton Rouge PP plant

March 1, 2019 — ExxonMobil Corp. (Irving, Tex.; www.exxonmobil.com) will fund the construction of a new polypropylene (PP) unit in Baton Rouge, La., which will expand production capacity along the Gulf Coast by up to 450,000 m.t./yr. Construction will begin in 2019 and startup is anticipated by 2021.

Ineos to build new vinyl acetate monomer plant in the U.K.

February 27, 2019 — INEOS (London; www.ineos.com) will invest £150 million in Hull, U.K. to construct a new vinyl acetate monomer (VAM) plant. The capacity at the site is expected to be 300,000 m.t./yr of VAM, a key component in glass, adhesives, coatings, films and more.

Air Liquide to construct world's largest proton-exchange membrane electrolyzer

February 25, 2019 — Air Liquide (Paris; www.airliquide.com) plans to construct the largest proton-exchange membrane (PEM) electrolyzer in the world, with a 20-MW capacity for the production of carbon-free hydrogen. Air Liquide will install the 20-MW electrolyzer unit to increase the current capacity of its hydrogen facility located in Bécancour, Canada by 50%.

Solvay to boost soda ash productivity with expansion; new biomass boiler

February 25, 2019 — In the next two years, Solvay S.A. (Brussels, Belgium; www.solvay.com) plans to undergo several measures to increase production capacity of soda ash by 500,000 m.t./yr and of sodium bicarbonate by 100,000 m.t./yr. Solvay is also investing in a new biomass boiler at its soda ash plant in Rheinberg, Germany, which will lower CO_2 emissions by 190,000 m.t./yr when the boiler comes onstream in May 2021.

Mergers & Acquisitions

Axens and ExxonMobil sign alkylation alliance agreement

March 12, 2019 — ExxonMobil and Axens (Rueill-Malmaison, France; www.axens.net) have signed an alliance agreement for the sublicense of ExxonMobil's Sulfuric Acid Alkylation technology. Under the agreement, Axens and ExxonMobil will market and deliver all technologies associated within a petroleum-refinery alkylation block, including Axens' feed preparation process, *n*-butane isomerization and ExxonMobil's Sulfuric Acid Alkylation. The technologies will be offered under a single license to be provided by Axens.

Evonik to sell its Methacrylates business for €3 billion

March 5, 2019 — Evonik Industries AG (Essen, Germany; www.evonik.com) has signed an agreement to sell its Methacrylates business to Advent International (Boston, Mass.; www.adventinternational.com) for €3 billion. The transaction comprises the Methacrylates, Acrylic Products and CyPlus business lines and some of the methacrylate resins activities, as well as 18 global production sites.

BP, Virent and Johnson Matthey partner on production of bio-based PX

March 5, 2019 — BP (London; www.bp.com), Virent Inc. (Madison, Wis.; www.virent.com) and Johnson Matthey (London; www.matthey.com) have signed an agreement that will further advance the commercialization of Virent's Bioforming process for production of bio-based *p*-xylene (PX), a key raw material for the production of renewable polyester. BP will contribute technical resources and has exclusive rights to negotiate becoming the sole manufacturer of bio-PX using Virent's technology.

Axens acquires a stake in biogas specialist Arol Energy

March 5, 2019 — Axens has taken an equity interest in Arol Energy (Le Bourget-du-Lac, France; www.arol-energy.com), an expert in biogas purification systems. The company's products and services are designed for entities involved in biogas production from urban or agricultural waste.

Messer and CVC finalize acquisition of selected Linde and Praxair assets

March 3, 2019 — Messer Group GmbH (Bad Soden, Germany; www.messergroup.com) acquired the majority of Linde AG's (Munich, Germany; www.linde.com) gases business in North America and certain Linde and Praxair (Danbury, Conn.; www.praxair.com) business activities in South America, in a joint venture (JV) with CVC Capital Partners Fund VII (CVC). The JV — called Messer Industries GmbH — is investing a total of around $3.6 billion (€3.2 billion). Messer is contributing the majority of its Western European companies to the JV.

Matheson completes acquisition of Linde's HyCO business in the U.S.

March 1, 2019 — Matheson Tri-Gas, Inc. (Bernards Township, N.J.; www.mathesongas.com) has completed its acquisition of the divested Linde HyCO business in the U.S., which produces H_2, CO and syngas. The business became available due to the global business combination of Linde and Praxair. The HyCO business includes a remote operations center in LaPorte, Tex. that supports plants in Alabama, Illinois, Ohio and Washington.

Total acquires French plastics recycler Synova

Total S.A. (Paris, France; www.total.com) has acquired Synova, a France-based manufacturer of high-performance recycled polypropylene for the automotive sector. Synova currently produces 20,000 m.t./yr of polypropylene from recycled plastics. ■

Mary Page Bailey

A Bright Future for Quantum Dots

The novel performance characteristics and tunability of quantum dots make them a promising nanomaterial in numerous emerging applications

IN BRIEF

FIRST CONTINUOUS PROCESSING METHOD

QDS FOR SUSTAINABILITY

ADVANCED IMAGING AND BEYOND

Quantum dots (QDs) represent a broad group of semiconducting nanoparticles that feature a unique combination of optical and electronic properties. For example, QDs provide many of the same benefits as organic dyes in existing applications, but they are more robust in terms of light-conversion capabilities and can also withstand harsh chemical solvents, higher temperatures and corrosion, while generally providing a broader light-absorption band. Perhaps QDs are most prominently known for their ability to emit extremely pure colors for long durations (Figure 1) — they have been widely used to improve the display characteristics of high-end televisions. But QDs' applicability stretches far beyond consumer electronics into wide-ranging potential end uses from solar power to agriculture to water purification.

Quantum Materials

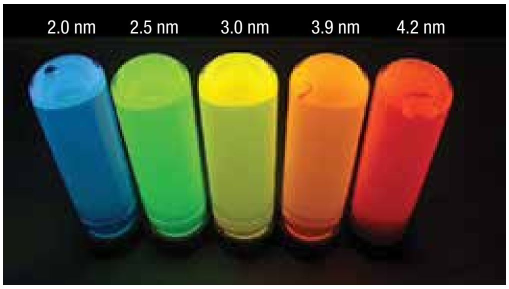

FIGURE 1. Quantum dots are known for their ability to instill brilliant colors into electronics displays, but their chemistry and tunability make them ripe for innovation in many other areas

First continuous processing method

Quantum Materials Corp. LLC (QMC; San Marcos, Tex.; www.qmcdots.com) has developed what is said to be the world's first continuous production process for QD manufacturing. QMC's technology uses all fluidized materials and continuous-flow reactors, whereas the vast majority of QD production depends on batch production. "We use microflow reactors, which can achieve high rates of heat and mass transfer, resulting in a very uniform product. Therefore, very little post-processing is required," explains Krishna Kowlgi, senior research engineer at QMC. In addition to product homogeneity, another benefit of continuous QD processing over batch is time — the entire continuous-flow process can be automated and requires much less manual intervention. "From start to finish, it takes a minimum of 5 s to a maximum of 19 min, based on the QD material type, to convert raw materials to functional QDs. We think this is the fastest QD production currently on the market. The numbers in literature for batch QD processes range from multiple hours to days or even weeks," adds Kowlgi. He emphasizes that QMC's reactors are flexible enough to produce different QDs for different end uses. "The light-emission capabilities of the QD depends on its geometry and chemical composition. A 4-nm spherical QD would produce different emissions than one with a 3-nm diameter," he adds. Due to the fluidized flow chemistry of QMC's process, QDs are produced in solution, so they are either filtered or undergo some other type of post-processing separation step.

Currently, QMC has production capabilities to manufacture around 3 kg/h of QDs at its site in San Marcos (Figure 2). In late 2018, the company signed a license agreement to construct a large-scale QD plant in Assam, India. QDs produced at the Assam site are expected to mainly be deployed in solar power and display and lighting applications. Once operational, the Assam site will be among the world's largest manufacturing plants for active nanoparticles, and the only industrial-scale site to employ continuous QD production.

Moving forward, QMC is continuing to push the boundaries of its continuous-flow process. "Because the flow reactors have a very small footprint, they are well suited for handling extreme process conditions," explains Kowlgi. By examining different combinations of processing conditions, more robust QDs can be tai-

lored for a broader range of end uses. Kowlgi lists anti-counterfeit inks (Figure 3) and tags, digital camera sensors and photovoltaics as important emerging application areas for QDs in the coming years.

QDs for sustainability

QDs also hold promise in improving environmental sustainability. UbiQD, Inc. (Los Alamos, N.M.; www.ubiqd.com) has developed specialized QDs for agricultural and solar energy applications. According to Hunter McDaniel, founder and CEO of UbiQD, the company's QDs bring together a unique semiconducting composition and a novel luminescence mechanism into a low-toxicity formula — based on zinc or copper rather than commonly used, more toxic materials, such as cadmium (Cd) or lead (Pb) — to enable QDs' use in applications that were not previously accessible at a commercial scale. "Our main focus is large-area applications that involve the manipulation of sunlight. Greenhouse agriculture and commercial building windows are our priorities. In both cases, we are partially absorbing sunlight, changing the color, and then re-routing that light energy to either boost crop yield or generate electricity." explains McDaniel. The UbiGro greenhouse-focused product line is commercially available, while the solar-window materials are still in the development phase. UbiGro is an active QD-based film used on greenhouses (Figure 4) that red-shifts the sun's spectrum. McDaniel says that UbiGro has shown effectiveness in improving yield and quality over a variety of climate conditions with many crops, including tomatoes, cucumbers, leafy greens, hemp and more.

FIGURE 2. QMC's continuous process for QD manufacturing enables a considerably faster conversion from feedstock to functional materials

UbiQD utilizes a liquid-phase batch reaction to make about 1 kg of QDs per 10 L of reactor each week. McDaniel says that the key to manufacturing the company's unique QDs is in the chemical recipes and post-processing steps, and that the process requires lower costs and results in higher-stability materials than other QD manufacturing processes.

Although QDs are still mainly thought of as display components,

Quantum Materials

FIGURE 3. An emerging application for QDs is in anti-counterfeit inks, where their unique color signatures can strengthen security measures

UbiQD believes that the agricultural market will quickly become another major application area, with solar energy not far behind. "Quantum dots are already making the world a better place, but there is huge untapped potential," emphasizes McDaniel.

Water purification is another area where QDs can improve sustainability. A group of researchers from the University of South Carolina (Columbia; www.sc.edu) and the SmartState Center of Catalysis for Renewable Fuels (www.smartstatesc.org) have successfully integrated QDs into a reverse-osmosis (RO) membrane designed for high-flux desalination processes. The team used nitrogen-doped graphene-oxide quantum dots (N-GOQDs) as an additive in the fabrication of a polyamide membrane. "The very small size of GOQDs and their rich functional groups make them an excellent additive in polymeric membranes, or even as basic building blocks for GOQD membranes if packed and crossed appropriately," says Miao Yu, one of the lead researchers on the project, and currently a professor of chemical and biological engineering at Rensselaer Polytechnic Institute (Troy, N.Y.; www.rpi.edu). According to Yu, this work is the first to investigate GOQDs in membrane applications. "There are many nanoparticles that have been added to RO membrane matrices, but much of this work is still at the lab stage, and the water flux increase is not as significant as that seen with the addition of N-GOQDs," he adds. The research showed that adding just 0.02 wt.% of GOQDs tripled the polyamide membrane's water permeability, while still rejecting salts at a comparable rate to an untreated polyamide membrane.

Yu says that N-GOQDs' functional groups can be easily crosslinked into an RO membrane matrix without introducing large cavities, and that their hydrophilic nature helps to further facilitate water transport, increasing flux. Thus far, a membrane with a permeation area of 10 cm^2 has been demonstrated, but Yu says that the fabrication process, based on interfacial polymerization, is compatible with industrial membrane-preparation processes, making its scaleup more promising. The team has applied for a patent and plans to work with an industry partner on larger-scale production.

Advanced imaging and beyond

In China, NajingTech (Hangzhou; www.najingtech.com) is working to extend the performance of QDs for both photoluminescent (photon-to-photon) and electroluminescent (electron-to-photon) uses. Founded in 2002 by QD pioneer and former professor of chemistry at the University of Arkansas, Xiaogang Peng, NajingTech's U.S. subsidiary, NN-Labs LLC (Fayetteville, Ark.; ww.nn-labs.com) was among the first commercial providers of QD nanocrystals. Currently, NajingTech is focused on the deployment of QD light-converting devices (QLCD) and QD light-converting films (QLCF), as well as commercializing electroluminescent QD light-emitting diode (QLED) technologies (Figure 5), along with emerging work in the fields of photovoltaics, sensors and detectors, lasers, bio-imaging and more, says Wei Huang, senior researcher at NajingTech. On the biotechnology front, NajingTech is currently working to develop QD-based immunochromatography technologies and QD-linked immunosorbent assays. The company is also investigating ways to minimize or eliminate the Cd content in QD materials for large-scale applications and electroluminescent

UbiQD

FIGURE 4. QD films in greenhouses can adjust sunlight to improve crop yield

devices, since Cd introduces toxicity and limits materials' ability to be used in certain commercial end markets. For example, NN-Labs has developed copper indium sulfide/zinc sulfide (CuInS/ZnS) core-shell QD products for use in biomedical, solar energy and LED lighting applications. The company also offers a line of indium phosphide/zinc sulfide-based (InP/ZnS) QDs as an alternative to Cd-based formulations.

"We use a solution-based 'green synthesis' method to make QDs by using the non-coordinating solvent octadecene along with stable and safe precursor materials," explains Huang. This synthesis method enables precise tuning of nanocrystal size and size distribution, and the use of a non-coordinating solvent is said to make the process simpler and more environmentally friendly than other techniques. Since being developed by Peng and patented in 2006, many QD companies have also adopted this process. In the future, says Huang, the company plans to deploy a printing-based technology to fabricate QLED-based technologies.

MilliporeSigma (Burlington, Mass.; www.emdmillipore.com) and EMD Performance Materials, two of the North American operating companies of Merck KGaA (Darmstadt, Germany; www.merckgroup.com), supply QD materials for researchers working on

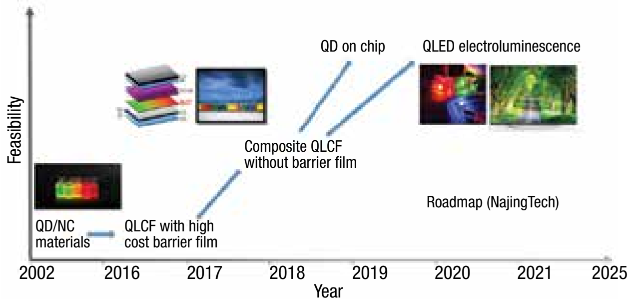

FIGURE 5. Refining electroluminescent properties is the next step in implementing QDs into more complex LED applications

photovoltaics, LEDs, lasers, bio-imaging and more. To facilitate such a wide range of end uses, ensuring consistency in surface chemistry and other functional properties is critically important. "Composition, size and geometry are all important factors that are customized to fit all of these diverse applications," says Bryce Nelson, head of materials science at MilliporeSigma. "For example, PbS-based QDs exhibit fluorescence in the infrared regime, while InP/ZnS and Cd-based QDs possess fluorescence emission in the ultraviolet-visible range. Adjustments in size allow the fine-tuning of fluorescence properties," he continues. And depending on the end market, further customization may be required, such as optimizing surface chemistry for compatibility with a surrounding polymer or matrix in display applications. The QD synthesis method is also critical in tailoring materials for new end-use applications — common techniques include the hot-injection method and the heat-up method, among many others.

"Solar cells and lasers come to mind as areas with a lot of research activity right now. For in-vivo applications, QDs with infrared emission capabilities enable enhanced single-cell imaging, as well as deep-tissue mono- and multi-photon imaging," says Nelson. However, he emphasizes that the use of QDs in-vivo is still hampered by challenges related to toxicity and lack of biodegradability, further highlighting the importance of reducing heavy-metal content. According to Nelson, the Performance Materials business of Merck is working toward Cd-free QDs for display applications, which can provide the increased color range of QDs without the toxicity of heavy metals. EMD Performance Materials is also currently developing new QD technologies for next-generation organic LED (OLED) displays, which will require complex chemical formulations to vastly improve color quality. While these new technologies, says Nelson, are still a few years from commercialization and significant formulation challenges remain, the combination of OLED and QD technologies has the potential to bring a new standard of performance in terms of brightness, efficiency and viewing angle.

From displays to biomedical imaging to solar energy and agriculture, the flurry of activity around tailoring QDs for ever-expanding application fields is certainly going to continue in the coming years, with an increasing number of novel end-uses expected to appear, as formulation and synthesis techniques are further honed. ■

Mary Page Bailey

Distillation: It's What's on the Inside that Counts

New column internals boost efficiency and improve reliability of distillation towers

IN BRIEF

IMPROVING EFFICIENCY AND RELIABILITY

ADDRESSING SPECIFIC CHALLENGES

Koch-Glitsch

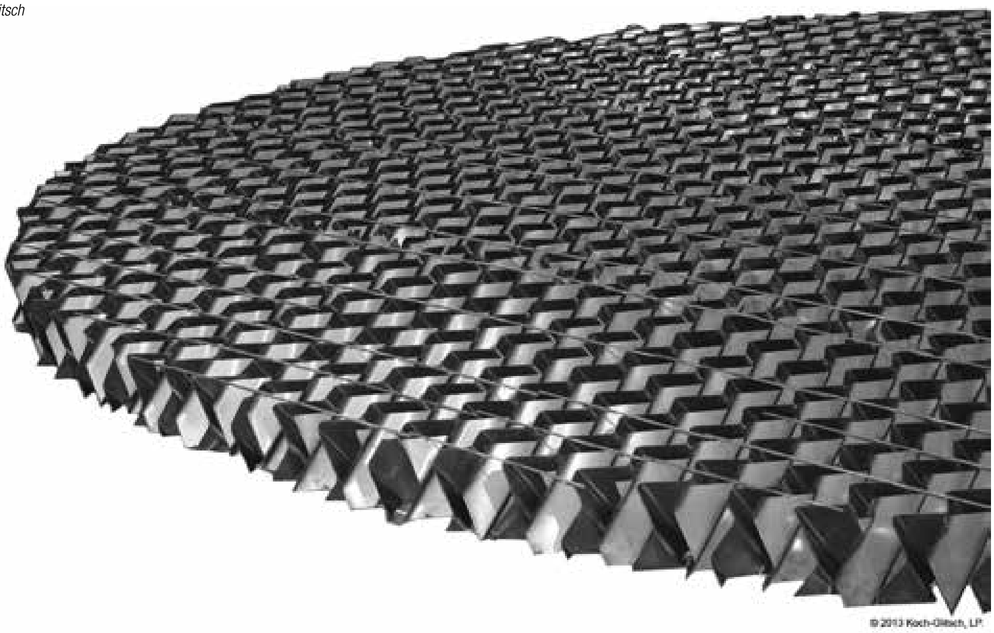

FIGURE 1. Proflux severe-service grid packing is a high-performance severe-service grid packing that combines the efficiency of structured packing with the robustness and fouling resistance of grid packing

In the chemical process industries (CPI), the majority of separations are done via distillation columns. And, when the rest of the process relies upon those columns, inefficiencies, bottlenecks and shutdowns are problematic. In an effort to keep distillation processes — and the rest of the plant — chugging along, column internals are being tweaked and re-worked to help optimize efficiency and reliability of the columns.

"Whether it is in refining, chemical processing or producing plastics, most of the separation between organic chemicals is being done with distillation. At the same time, there is a constant pressure for chemical processors to make their processes more cost effective," says Izak Nieuwoudt, chief technical officer with Koch-Glitsch (Wichita, Kan.; www.koch-glitsch.com). "Because distillation columns are a huge energy consumer and because people don't want to spend a lot of time fixing equipment, increasing efficiency and reliability of the columns is at the forefront right now."

Often after a process is up and running, processors find that the energy consumption is much higher than they were expecting, says Antonio Garcia, mass transfer business development manager with AMACS Process Tower Internals (Arlington, Tex.; www.amacs.com). "To obtain better energy efficiency, they must explore their options to improve the mass-transfer performance," he says. "In addition, processors are often looking for ways to de-bottleneck the process in order to obtain better separation and capacity requirements and fouling is a common cause of bottlenecks, so finding technologies that assist with these issues is also important."

Bottlenecks and downtime caused by fouling or mechanical issues, such as vibration or mechanisms within the columns coming apart, can become very costly. "It's very expensive every time you have to shut down a distillation column, because it often results in the shutdown of upstream and downstream units, as well," says Nieuwoudt. "And, these unplanned shutdowns

RVT Process Equipment

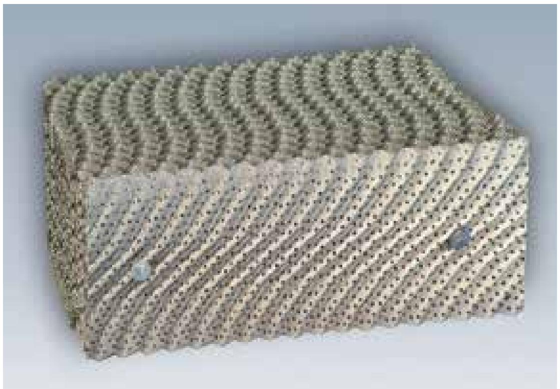

FIGURE 2. A new, high-capacity structured packing, the SP-Line from RVT, offers modified channel geometry, lower pressure drop and higher capacity

RVT Process Equipment

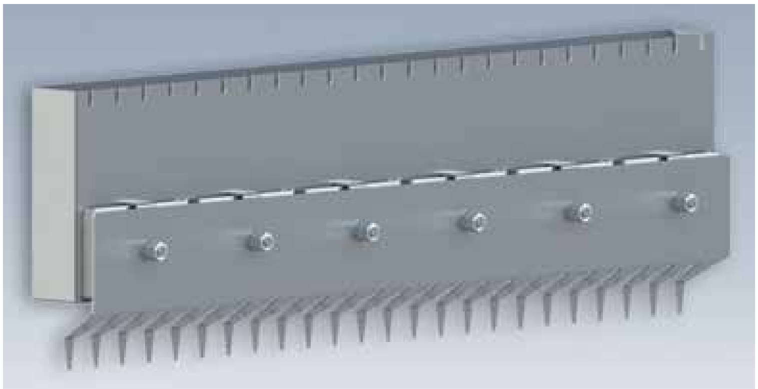

FIGURE 3. For very low liquid loads, another application-specific challenge, packings can be combined with new types of liquid distributors

result in large losses per day."

For this reason, manufacturers of column internals are developing products designed to aid processors in increasing energy efficiency and reliability.

Improving efficiency & reliability

Replacing conventional trays and packings with newer, advanced solutions is often necessary for a processor who is seeking higher efficiency, capacity and reliability, so manufacturers are constantly looking to improve upon their offerings.

For instance, Raschig GmbH (Ludwigshafen, Germany; www.raschig.com) has recently released the Raschig Super-Ring Plus, a new, high-performance random packing that exceeds the performance of the previous Raschig Ring. "The optimized structure of Raschig Super-Ring Plus enables a further capacity increase at constant efficiencies," says Micheal Schultes, technical director at Raschig. "The product is the result of design development based on many years of research. The target was to stay with all the advantages of the Super-Ring, but improve capacity and reduce pressure drop."

The resulting product minimizes

pressure drop by arranging flat sinusoidal strips into an extreme open structure, maximizes capacity by film flow preference on continuous sinusoidal-strip arrangements, increases efficiency by minimizing droplet formations inside the packing and decreases fouling tendency by reducing droplet development and offering low pressure drop. Fouling sensitivity is also reduced by generating continuous liquid films, wetting the entire packing element.

Likewise, AMACS has been doing research to improve its SuperBlend product. "Research has shown that by replacing existing random packing with our SuperBlend 2-PAC, tower efficiency can be increased by 20% or capacity by 15%," says Moize Turkey, manager, applications engineering, with AMACS. The SuperBlend 2-PAC technology is a blend of high-performance packing sizes placed in a single bed. "We blend two sizes of the best metal random geometry and, when combined, the patented blend achieves the efficiency benefits of the smaller packing size, while retaining the capacity and pressure drop of the larger packing size," he says. The blended bed is recommended for absorption and stripping, fine chemical distillation, refinery fractionators and retrofit opportunities in any mass- or heat-transfer tower limited by conventional or third-generation random packing.

Improvements to internals are also being developed to assist with issues such as fouling and difficult conditions.

"Reliability is extremely important for day-to-day considerations. No matter how well a device performs, if it can't stand up to the fouling conditions in a process, it won't be successful," says Mark Pilling, manager of technology USA with Sulzer

DeDietrich

FIGURE 4. DeDietrich focuses on columns and internals for highly corrosive processes at temperatures up to 390°F. Columns up to DN1000 are made of QVF borosilicate glass 3.3 or DeDietrich glass-lined steel. Bigger columns up to DN2400 are made of DeDietrich glass-lined steel only. The corrosion-resistant materials are made of borosilicate glass 3.3, SiC, PTFE or tantalum

(Winterthur, Switzerland; www.sulzer.com). "Sulzer has spent a tremendous amount of time over the last five years developing a complete line of fouling-resistant equipment." In trays, the company offers VG AF and anti-fouling trays, and recently launched UFM AF valves, which are both high performance for capacity and efficiency, as well as extremely fouling resistant. In packings, the company launched Mellagrid AF anti-fouling grid packings, which are suitable for highly fouling packing applications, such as vacuum tower wash sections.

Pilling adds that for foaming issues, Sulzer has been working on a two-pronged approach. "While we develop equipment and designs to handle foaming applications, we also work with our customers to determine potential foaming applications," he says. "Once you know foaming exists, you can design for it. It's the cases where a customer will have a foaming condition and not know about it that tend to create problems. We see all sorts of foaming, such as Marangoni, Ross foams and particulate foams and work with customers to identify such situations."

And, for applications where fouling and coking can be very severe, Koch-Glitsch developed Proflux severe-service grid packing, says Nieuwoudt (Figure 1). The new high-performance severe-service grid packing combines the efficiency of structured packing with the robustness and fouling resistance of grid packing. It is an assembly of sturdy corrugated sheets welded to heavy-gauge rods. The combination of welded rod assembly and corrugated sheets of increased material thickness provides a robust design that resists damage from tower upsets or erosion. The gaps between the sheets provide improved fouling resistance. "The packing has been installed almost 100 times now in very severe-fouling services and is really doing well compared to the products it is replacing. The longer run life and the lower pressure drop it provides results in lower operating costs for the customer," says Nieuwoudt.

Addressing specific challenges

When it comes to distillation, there are also often challenges specific to a process that need to be addressed through special measures.

"There is a market for tailor-made solutions that are tuned to the specific process and customer needs," says Christian Geipel, managing director, with RVT Process Equipment (Steinwiesen, Germany; www.rvtpe.com). "This is especially valid for revamps of existing plants that are modified to fulfill new demands. The challenges are various and include objectives such as longer and more predictable run lengths for fouling applications, higher capacity and lower pressure drop or wider operating ranges for more flexibility."

To address specific needs, RVT has developed a high-capacity structured packing, the SP-Line (Figure 2). "Due to modified channel geometry, lower pressure drop and higher capacity are achieved." Further, for very low liquid loads, another application-specific challenge, these packings can be combined with new types of liquid distributors. "An improved spray nozzle distributor that combines spray nozzles with splash plates was developed and is successfully used in applications like refinery vacuum columns," says Geipel. "It reduces entrainment and therefore fouling in the packing sections above the distributor without sacrificing liquid distribution quality to the packing section below."

Another new liquid distributor from RVT (Figure 3) is a trough-type dis-

GTC Technologies

FIGURE 5. Towers that have a side cut, but are thermally inefficient, may be good candidates for dividing-wall column technology

tributor with splash plates that combines low liquid rates with a higher operating range and a robust, fouling-resistant design.

Similarly, GTC Technology US, LLC (Houston; www.gtctech.com) is developing new products to assist processors with improving the performance of distillation columns based on their specific needs. One of the latest developments includes GT-OPTIM high-performance trays, says Brad Fleming, general manager for GTC's Process Equipment Technology division. Hundreds of industrial installations plus testing at Fractionation Research Inc. (FRI; Stillwater, Okla.; www.fri.org) have demonstrated that the high-performance tray achieves significant efficiency and capacity improvement over conventional trays. The cross-flow trays are customized to the end user's needs to achieve high efficiency via a combination of patented and proprietary devices that make up each tray design. "We can provide a collection of technologies and features that can be employed in order to address specific objectives," notes Fleming. "One processor's objective might be to increase efficiency, while another wants to increase capacity and still another wants to minimize pressure drop, mitigate fouling or extend runtime. We have many different weapons in our equipment design arsenal, so we are able to focus on the customer's targeted objective for their specific process improvement."

Meanwhile, AMACS has addressed another common distillation challenge faced by petroleum refineries, petrochemical plants, gas plants and similar facilities. Often, a vertical knockout drum or separator with mist-elimination equipment installed fails to remove free liquid from a process gas stream. "Instead of trying to address or repair symptoms, we look for the root cause, which usually involves the mist-elimination equipment in the knockout drum," says AMACS's Garcia. To address the problem, the company developed the Maxswirl Cyclone, a high-capacity, high-efficiency mist-elimination device that uses centrifugal forces to provide state-of-the-art separation performance.

The Maxswirl Cyclone tubes consist of a fixed swirl element, which applies centrifugal force on mist-laden vapor to separate entrained liquid from gas flow. In this axial-flow cyclone, the resulting centrifugal force pushes liquid droplets outwards, where they create a liquid film on the cyclone inner wall. The liquid passes through slits in the tube wall and gets collected at the bottom of the cyclone box and drained by gravity. The dry gas concentrates in the center of the cyclone tube and exits through the cyclone.

Meanwhile, DeDietrich (Mainz, Germany; www.dedietrich.com) is focusing effort on providing columns and internals for highly corrosive processes at temperatures up to 390°F, says Edgar Steffin, head of marketing with DeDietrich. "Columns up to DN1000 are made of QVF borosilicate glass 3.3 or DeDietrich glass-lined steel. Bigger columns up to DN2400 are made of DeDietrich glass-lined steel only. The corrosion-resistant materials are made of borosilicate glass 3.3, SiC, PTFE or Tantalum" (Figure 4).

He adds that most processes at elevated temperatures above 300°F require the avoidance of PTFE. SiC has a higher temperature resistance and permits the design of bigger distributors and collectors that are less sensitive for feeds containing

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solids or those tending to foam, degas or to flash.

The company's Durapack structured packing in borosilicate glass 3.3 is suitable for corrosion-resistant glass 3.3 or glass-lined steel columns, as it has the same corrosion resistance as the glass column and keeps its thermal stability at higher temperatures compared to polymers. Borosilicate glass 3.3 is nonporous, which substantially cuts erosion and corrosion compared to equivalent ceramic packing.

And, towers that have a side cut, but are thermally inefficient, says GTC's Fleming, may be good candidates for dividing-wall column technology. "Many distillation columns have a top and bottom product, as well as a side-draw product, but with this comes a lot of thermal inefficiency. Dividing-wall column technology — where you revamp the traditional column — is one way to increase capacity while reducing energy consumption or reducing the yield impurity of the products," he says (Figure 5).

The dividing-wall column separates a multi-component feed into three or more purified streams within a single tower, eliminating the need for a second column. The design uses a vertical wall to divide the middle of the column into two sections. The feed is sent to one side of the column, called the pre-fractionation section. There, the light components travel up the column, where they are purified, while the heavy components travel down the column. The liquid flow from the column's top and the vapor flow from the bottom are routed to their respective sides of the dividing wall.

From the opposite side of the wall, the side product is removed from the area where the middle-boiling components are most concentrated. This arrangement is capable of producing a much purer middle product than a conventional side-draw column of the same duty, and at higher flowrate.

"The conversion to a dividing-wall column is investigated when you're looking at making significant improvements you couldn't do otherwise within the constraints of a traditional tower, but if you can convert to dividing-wall technology, you will see significant decrease in energy consumption," he says. "Generally, there is a 25 to 30% reduction in overall energy consumption for a given throughput, dramatically improved yield and purity of products and often an increase in throughput, as well."

He adds that there is also opportunity to use a dividing-wall column to replace a traditional two-tower sequence. "You can use dividing-wall columns to perform the same operation and produce the same products, but you're doing it in one physical tower in comparison to a two-tower scheme. In the grassroots realm, a substantial reduction in capital expenditures can be achieved with dividing-wall column technology." ■

Joy LePree

Focus on Analyzers

Dynatrol

Spectro Analytical Instruments

Electro-Chemical Devices

A proportional level-output detector for pilot plants

The Dynatrol CL-10GP proportional level detector (photo) is designed to control liquid levels in pilot plants, chemical-processing plants and small vessels, or anywhere it is necessary to obtain proportional level control over a precise range. The EC-103C(G) control unit is paired with the detector and can activate electro-pneumatic transducers, valve positioners, indicators, controllers or other direct-current devices. The detector and control unit accurately monitor and control an extremely precise liquid level range due to its unique, high-resolution, proportional output signal. This unique control operates reliably under varying-frequency power supplies or harsh process conditions, such as high pressures and high temperatures. All units are built in accordance with Class I, Group D; Class II, Groups E, F & G; and Class III services. — *Automation Products, Inc.; Dynatrol Div., Houston*
www.dynatrolusa.com

Determine sulfur and nitrogen content in gases and liquids

Launched under this company's Antek brand, ElemeNtS is said to be the most advanced modular-analytical system available for the detection of total nitrogen or total sulfur (or both) content in liquid or gaseous materials and liquified propane gas (LPG) samples. Three configurations are available: ElemeNtS-N for nitrogen analysis, ElemeNtS-S for sulfur analysis and ElemeNtS-N/S for simultaneous nitrogen and sulfur analysis. ElemeNtS combines extremely stable sulfur and nitrogen detectors with linearity up to 10^4. The new generation ElemeNtS displays exceptional sensitivity and allows simultaneous analysis of total sulfur and total nitrogen in hydrocarbon samples up to 30 parts per billion by weight (ppbw), according to ISO 11843. Featuring a 10 in. touchscreen, the ElemeNtS allows for direct queue control and diagnostics, along with a completely revamped IRIS-based software platform. ElemeNtS complies with key standard methods. — *PAC LP, Houston*
www.paclp.com

This ICP-OES analyzer is now twice as sensitive

The new Spectrogreen inductively coupled plasma, optical-emission spectrometry (ICP-OES) analyzer (photo) features a revolutionary Dual Side-On Interface (DSOI) technology that achieves twice the sensitivity of conventional radial-plasma-view instruments, at an attractive price/performance ratio. DSOI technology, a brand-new approach to the critical issue of plasma view design, uses a vertical plasma torch, observed via a new direct radial-view technology. Two optical interfaces capture emitted light from both sides of the plasma, using only a single extra reflection, for added sensitivity and elimination of issues that plague newer vertical-torch dual-view models. As a result, DSOI provides twice the sensitivity of conventional radial systems and yet avoids the complexity, drawbacks and cost of vertical dual-view models. — *Spectro Analytical Instruments GmbH, Kleve, Germany*
www.spectro.com

A cyanide analyzer for industrial wastewater

The S80-T80 cyanide analyzer monitoring system (photo) features an S80 Pion Cyanide Sensor and a dual-channel T80 Transmitter. The cyanide analyzer helps plants achieve a more cost-effective cyanide-removal water-treatment system, which ensures wastewater is treated to meet U.S. Environmental Protection Agency (EPA) regulatory requirements prior to effluent discharge. The S80's CN-ion electrode is a combination electrode with a silver cyanide/silver sulfide solid-state pressed-crystal sensing element and a double junction reference electrode. The CN-ion selective electrode cartridge develops a millivolt potential proportional to the concentration of free CN ions in the mea-

Note: For more information, circle the 3-digit number on p. 58, or use the website designation.

sured solution. The typical output is 54–60 mV per decade of change in concentration. The sensor's response time varies from a few seconds in concentrated solutions up to a few minutes in the lower parts per million (ppm) range. This analyzer measures CN from 0.2 to 260 ppm. — *Electro-Chemical Devices (ECD), Anaheim, Calif.*
www.ecdi.com

Limit-value display simplifies process monitoring

LiquiSonic systems (photo) measure concentration and density in process liquids by sonic velocity. In addition to displaying the numeric concentration, density and temperature, the LiquiSonic now also displays the limit-value ranges, which simplifies the monitoring of an approximation of critical values. Users can identify the segment in the measuring range where the current value is settled on and get a quick overview. The LiquiSonic controller display allows up to three value ranges at the same time. This applies for each one of the up to four sensors, which can be connected to a control unit. — *SensoTech GmbH, Magdeburg–Barleben, Germany*
www.sensotech.com

New functionality for this sensor transmitter

This company has added the next-generation Signet 9950 dual channel transmitter (Gen 2b) to its flow and analytical instrumentation product line. The 9950 Gen2b adds extensive new functionality to its already proven capabilities that enable multiple measurement readings in one transmitter. This minimizes the number of transmitters needed, providing cost savings and greater convenience. The 9950 Gen 2b fully supports the Signet 2751 pH/ORP Smart Sensor Electronics for pH/ORP measurements. Additionally, the 9950 Gen 2b automatically measures pH glass impedance, detects broken glass and reads stored data in all Signet DryLoc pH and ORP electrodes. The 9950 Gen 2b also supports two new plug-in modules (photo): the Single Channel Direct Conductivity Module (3-9950.394-1) and the Dual 4–20-mA Current Loop Output Module (3-9950.398-2). — *GF Piping Systems, Irvine, Calif.*
www.gfps.com

Moisture analyzer with more communication capabilities

This company has broadened its AMEVision communication and display system capabilities to include the 3050 Series of moisture analyzers (photo). Originally designed for WDG-V analyzers, the AMEVision system provides realtime moisture measurements and trend data while also detailing a variety of maintenance and troubleshooting information. Users can now continually monitor the measurement and diagnostic information for up to eight 3050 Series moisture analyzers. AMEVision enables the convenience of on-site calibration and communication with analyzers via Modbus RS485 and Ethernet LAN. — *Ametek Process Instruments, a unit of Ametek, Inc., Pittsburgh, Pa.*
www.ametekpi.com

An analyzer for residual gases in vacuum systems

The Leyspec (photo) is designed for efficient residual-gas analysis in high and ultra-high vacuum systems. Due to its compact size and flexible mountability, the device is suitable for very different installation situations and measuring tasks in research and industry. Equipped with an integrated display, users can display the partial pressures of the relevant gases at any time. If the user is interested in an additional gas in his or her process, another channel can be individually assigned to it. The intelligent software offers users various analysis options from which they can choose. Additional test procedures and functionalities are pre-installed, such as the helium leak test or the setting of warnings and error limits for certain gases. — *Leybold GmbH, Cologne, Germany*
www.leybold.com

A transmitter for analytical sensors where space is limited

The Liquiline compact CM82 transmitter (photo, p. 26) accepts pH, ORP, pH/ORP, conductivity, oxygen and chlorine sensor signals from this company's Memosens sensor platform. Its housing is only 11 cm long and 2 cm wide, so even combined with the sensor, it fits into almost every assembly, says the company. Although small, it is a fully developed

SensoTech

GF Piping Systems

Ametek

Leybold

Endress+Hauser

Horiba Scientific

Metrohm

E Instruments International

multiparameter transmitter, with access available via 4–20-mA HART, or Bluetooth from any iOS or Android device. The CM82 reads all sensor and calibration data stored in the head of a Memosens sensor. As a result, the sensor is automatically detected within seconds, and the measurement is immediately ready for use after a sensor change. — *Endress+Hauser, Greenwood, Ind.*
www.us.endress.com

Add spectroscopy to any standard microscope

With its unique set of accessories, the new Standard Microscope Spectroscopy (SMS) family of systems (photo) enable any standard microscope to be fitted with a spectrometer and a detector, thereby offering the ability to perform techniques such as Raman, steady-state and time-resolved photoluminescence, reflectance/transmittance, electroluminescence, photocurrent and dark-field scattering. The SMS platform brings unprecedented flexibility and modularity to performing spectroscopy on standard microscope systems, all without compromising the imaging functionality of the microscope. "Multitask your Microscope" is the theme behind these SMS systems. — *Horiba Scientific, Div. Horiba Instruments, Inc., Piscataway, N.J.*
www.horiba.com/scientific

Attachments for this handheld Raman system

The new Mira Flex package (photo) permits customization of the Mira handheld Raman analyzer for unique applications. The basic package includes a Mira device, USB cable, calibration standard and laser safety glasses. Users can then simply select the available Smart Attachments and accessories that meet their specific sampling needs. For sampling through a container, the Working Distance Attachments (SWD, LWD, XLWD, or UA) can handle everything from direct contact to 22-mm glass containers. For determining the toxicity of an unknown material, the Standoff Attachment enables safe sampling from a distance. Checking the formulation of a tablet is performed with the Tablet Holder. The Ball Probe Attachment allows sampling by direct contact, for instance, in a barrel where a proper focus is not required. Mira is a fast, easy, reliable and flexible solution for identification and verification of materials. — *Metrohm AG, Herisau, Switzerland*
www.metrohm.com

A cross-pipe version of this TDL gas analyzer

With most tunable diode laser (TDL) gas analyzers, loss of alignment leading to loss of measurement signal during a running process can be a constant problem. The Cross-Pipe GPro 500 analyzer means maintaining alignment is no longer an issue. The GPro 500 series of TDLs solves the alignment issue by combining both the source and the sensor into a single unit. A probe attached to the sensor features a corner cube at its end that redirects the laser beam back to the sensor; therefore, loss of alignment is not possible. The probe concept is highly reliable in stacks with a diameter of less than 1 m. For wider stacks, where gas concentration is not constant, the company has introduced the Cross-Pipe GPro 500, thereby extending the application coverage of its GPro 500 family. A newly developed, two-dimensional corner cube array is positioned in the pipe, opposite the analyzer head. Exact alignment is not required. — *Mettler-Toledo Process Analytics, Inc., Bellerica, Mass.*
www.mt.com

Analyze industrial combustion gas with this handheld device

The E1500 handheld combustion emissions analyzer (photo) is a rugged unit with the ability to measure carbon monoxide and oxygen from high-efficiency and condensing boilers, burners, engines, turbines, kilns, furnaces, incinerators and other industrial combustion processes. Featuring a new, large color display and expanded internal memory, the E1500 lets users see and save sample data without worrying about running out of memory. The E1500 also features pre-calibrated, field-replaceable sensors that allow for easy diagnostics and replacement to reduce downtime and costly repair charges. — *E Instruments International, Langhorne, Pa.*
www.e-inst.com

Gerald Ondrey

New Products

Krohne Messtechnik

Casella Solutions

Griffco Valve

Pepperl+Fuchs

New options for this biogas flowmeter

The Optisonic 7300 Biogas ultrasonic flowmeter (photo) for the measurement of dry and wet (raw) biogas with variable composition has been updated. Besides the standard temperature sensor, it now features an optional pressure sensor, which is mounted on the flow tube. In combination with the integrated flow computer, the additional sensor provides for advanced biogas measurement. A temperature sensor in combination with the measured velocity of sound enables direct measurement of methane content via calculation of the molar mass. By using an additional pressure sensor, Optisonic 7300 Biogas devices can also provide calculation of gas flow volume in standard conditions. The meter is available in line sizes 2, 3, 4, 6 and 8 in. (DN50, 80, 100, 150 and 200). — *Krohne Messtechnik GmbH, Duisburg, Germany*
www.krohne.com

This boundary monitor now includes VOC emissions data

The new Guardian2 site boundary monitor (photo) is designed to help site management remain compliant with emissions levels. Using remote monitoring and reporting of noise, dust and vibration levels, the Guardian2 now includes monitoring of volatile organic compounds (VOCs) at concentrations up to 6,000 parts per million (ppm). Equipped with a photoionization detector (PID), the Guardian2 generates text or email alerts when limits are exceeded and produces manual or automated reports for site dust or noise compliance. — *Casella Solutions, Bedford, U.K.*
www.casellasolutions.com

Backpressure valves and pulsation dampeners combined

New Fusion dampeners (photo) combine the technologies of this company's backpressure valves and pulsation dampeners to simplify a chemical-feed system installation by blending two previously separate accessories into one product. The combination of these two devices into one accessory will also reduce the number of joints, creating fewer leak-path opportunities and provide for a smoother, nearly pulseless chemical-feed system, says the company. The initial offering of Fusion dampeners is available in NPT connection sizes of 1/4, 3/8 and 1/2 in. and with two pulsation-dampeners in 6 and 10 in.^{3} volumes. Due to the large internal passages in the new fusion dampeners, there are no restrictions on flowrate or pumped fluid cleanliness, according to the company. The new devices have a maximum operating pressure of 150 psi (10 bars) at 70°F (21°C). — *Griffco Valve, Inc., Amherst, N.Y.*
www.griffcovalve.com.

A tablet makes data available, even in hazardous zones

The new Tab-Ex 02 industrial tablet (photo) is ATEX/IECEx Zone 1/Div. 1 certified and makes data permanently available across the entire process chain of a plant. Based on the latest Samsung Galaxy Tab Active, the Tab-Ex 02 also includes augmented reality (AR) capabilities to support the precise completion of tasks and ensure rapid plant commissioning and effective preventive maintenance and asset management. AR visualization, along with the permanent availability of all data for a specific plant or asset, allows users to quickly and easily identify objects, space requirements or technical defects. The Tab-Ex 02 also simplifies data exchange — when an entry is made, the date of modification and the employee's name are stored automatically. This guarantees the traceability of the data. No information is lost because it is entered directly via the mobile device. — *Pepperl+Fuchs GmbH, Mannheim, Germany*
www.pepperl-fuchs.com

A new digital service to transform engineering in the CPI

This company's Advanced Modelling and Simulation (AMS) service can help engineers to make better-informed decisions at an earlier stage, maximizing efficiency and reliability, reducing risks and optimizing operating costs (OPEX). Users can now virtually visualize heat- and fluid-flow scenarios that were merely speculated before. Building on the latest digital techniques and computer-aided

engineering (CAE) solutions, the AMS service is enabled by the powerful fluid-flow simulation platform TransAT, which delivers multi-dimensional, transient flow-process predictions. TransAT is a versatile fluid-flow simulation platform using smart technology for multi-dimensional meshing, and is particularly suitable for multiphase flows with complex physics. Furthermore, advanced simulations are, in some critical cases, the only way to understand key phenomena that control the processes, says the company. — *Pöyry Oyj, Vantaa, Finland*
www.poyry.com/ams

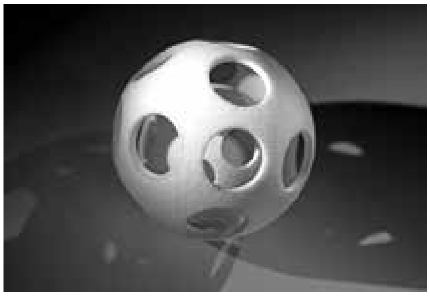
These flow controllers support many user-created mixtures

Aalborg Instruments

The DPC Series of precision digital mass-flow controllers for process gases (photo) provides simultaneous display of mass flow, volumetric flow, pressure and temperature parameters in scientific and analytical applications, bioreactors and surface depositions, gas sampling, manufacturing and metrology activities. DPC differential-pressure mass-flow controllers feature totalizers with batch processing mode, 200:1 turndown ratio and less than 150 ms response time. The devices' multi-gas functionality supports 90 different gases and gas mixes, and the user-defined mixture functionality allows for the creation of up to 20 custom gas mixes with up to five different gas components in each mixture. — *Aalborg Instruments, Orange, N.Y.*
www.aalborg.com

New polymer powder for 3-D printing

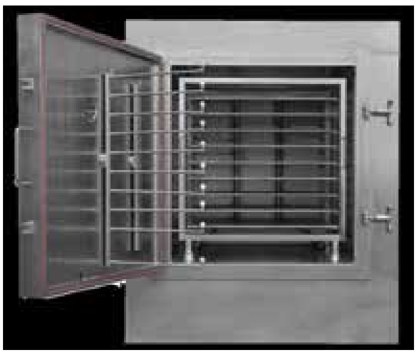
Evonik Industries



This company has developed a new polymer powder for applications in a higher-temperature range as part of its polyamide 6 series. The product further expands the company's portfolio of high-performance materials for powder-based 3-D printing technologies. The new polyamide powder features high mechanical strength, as well as excellent chemical and temperature resistance. Its heat deflection temperature (HDT B) is around 195°C. Moreover, the powder material stands out for its low water absorption — below 3% — which has a positive effect on processability in 3-D printing and the dimensional stability of printed 3-D components (photo). The new polymer powder has a nearly round grain shape, im-

ITT Goulds Pumps

Krüss



Italvacuum

proving the flowability, which makes it suitable for all powder-based 3-D printing technologies. — *Evonik Industries AG, Essen, Germany*
www.evonik.com

New pumps with a patented cyclone seal chamber

This company has expanded its ISO Series of process pumps with the addition of the ICO Open-Impeller i-Frame pump (photo). This new pump is ideal for handling gases and air, and is fully compliant with both ISO2858 and ISO5199 standards. Available in 34 sizes, the ICO pump includes a patented cyclone seal chamber, i-Frame power-end for improved service life and the i-Alert 2 equipment-health monitoring sensor as standard. New ICO pumps can handle flowrates as high as 450 m^3/h and heads up to 160 m. The operating temperature range is –40 to 280°C. Available materials of construction include carbon steel, 316 stainless steel, Duplex stainless steel, Alloy 20, Hastelloy, nickel and titanium. — *ITT Goulds Pumps, Seneca Falls, N.Y.*
www.gouldspumps.com

Measure roughness directly in the production process

Whether a surface must be very smooth or show a certain texture or degree of roughness, the new Surface Roughness Analyzer (SRA; photo) precisely pictures the topographic condition of a surface and provides information for optimizing the material or production process. Contactless, quickly, and with an extremely high resolution, the SRA delivers a 3-D image of a sample's surface and the correlating data to exactly describe its topography. Analyses with the SRA also help to evaluate the contribution of roughness to the wettability of a sample or to the adhesion of coatings. The SRA uses the confocal microscopy technique to create the spatial representation of the surface. This happens by stacking layers of 2-D images, each one with a very small depth of focus, while lowering the optics with extremely tiny increments, leading to a height resolution down to 10 nm. — *Krüss GmbH, Hamburg, Germany*
www.kruss-scientific.com

Universal control extends security to individual devices

The Xage Enforcement Point (XEP) platform extends network security to individual devices to deliver universal access control for industrial operations. XEP enables, for the first time, role-based access control and single sign-on for every device, ranging from legacy control systems to brand-new industrial internet of things (IIoT) machines — even those previously lacking any access-control protection. The XEP platform works with this company's tamper-proof security fabric, which is deployed at onsite gateways to protect multiple legacy and IIoT devices at once, providing access control, password rotation and managed interactions. Edge-to-cloud monitoring gives administrators visibility into and control of all access attempts, whether successful or not. Any attempts to unplug or bypass the XEP generate management alerts in the fabric. XEP ensures that individual industrial devices, if ever compromised, are isolated individually, rather than in a group of other critical, uncompromised assets. — *Xage Inc., Palo Alto, Calif.*
www.xage.com

This vacuum-tray dryer features a unique CIP system

The Model E1S fixed-shelf vacuum-tray dryer (photo) has a chamber volume of 1,150 L (40 ft^3) and includes nine heated and cooled shelves with 36 removable trays. Available materials of construction include stainless steel types 304, 304L, 316 and 316L, as well as Hastelloy and other alloys. In addition to the heated shelves, the E1S is jacketed on all sides, top and bottom, as well as the door. Many optional features are available, including the patented "Multispray" clean-in-place (CIP) system and nitrogen-sparging manifold. The Model E1S can be supplied with this company's Saurus piston-type vacuum pump, which can produce an ultimate vacuum level of 0.03 mbar, or the dryer can be used with other conventional vacuum-pump technologies. — *Italvacuum S.r.l, Turin, Italy*
www.italvacuum.com

Remote tank-level monitoring with cellular connectivity

ATEK Access Technologies

TankScan TSU1000 (photo) is an IIoT-enabled ultrasonic tank-monitoring system with 4G LTE cellular connectivity to provide remote level monitoring of deployed tanks, totes and containers, effectively decreasing the need for manual inspections at remote tank sites. It also allows material managers to proactively know when tanks need to be serviced. The non-contact TSU1000 ultrasonic level sensor is fully integrated into the battery-powered device, which can be installed in locations where network power and connectivity infrastructure are not available. The monitor has a durable weather-proof enclosure and long battery life, says the manufacturer. — *ATEK Access Technologies, LLC, Eden Prairie, Minn.*
www.tankscan.com

A dense-phase conveying system for sensitive products


Gericke USA

The Pulse-Flow PTA dense-phase pneumatic conveying system (photo) directs air or nitrogen into the pressure vessel and pipeline in timed, regular pulses that form the dry material into separate, wave-like, pulsed slugs. Creating a gentle conveying action, the Pulse-Flow PTA operates at high pressures with low gas velocities to safely convey sensitive products, using mass flow, up to 500 ft into a hopper, reactor vessel, silo, railcar or other receiving destination with minimal particle degradation or separation. Suitable for conveying fragile, free-flowing and cohesive powders, granulates and coarse-grained products, the automated Pulse-Flow PTA conveying system encompasses the feeding hopper, pressure vessel, pipeline, conveying and secondary air supply, as well as the company's computer-driven, touchscreen control system. — *Gericke USA, Inc., Somerset, N.J.*
www.gerickeusa.com

This magnetic separator is automatically cleaned

Introduced at Powtech this month, the Easy Clean magnetic separator is used to remove metal particles (larger than 30 µm) from powders in the food, chemical, ceramics and plastics industries. The Easy Clean flow magnet is notable for its high magnetic-flux density of over 12,000 Gauss on the bars that come into direct contact with the product. The magnet is dustproof to an overpressure of 1.5 bars and includes a simple controller. Cleaning requires 6 bars air and a 24-V d.c. start signal. Captured ferrous materials land in a collection tray. — *Goudsmit Magnetics Group, Waalre, the Netherlands*
www.goudsmitmagnets.com ■

Mary Page Bailey and Gerald Ondrey

Industrial Drying: Convection versus Conduction

Department Editor: Scott Jenkins

Adjustment and control of moisture levels in solid materials is a critical aspect in the manufacture of many chemical products. Drying can be defined as the vaporization and removal of water or other liquids from a solution, suspension, or other solid-liquid mixture to form a dry solid. A complicated process involving simultaneous heat and mass transfer, accompanied by physicochemical transformations, industrial drying is often accomplished through one or more of four broad mechanisms, including the following: direct drying (convection); indirect or contact drying (conduction); radiant drying; and dielectric or microwave drying. This one-page reference focuses on the differences between convection and conduction drying.

TABLE 1. CONVECTION VERSUS CONDUCTION DRYING PROPERTIES		
Property	**Convection**	**Conduction**
Carrier gas	Uses sensible heat of gas that contacts the solid to provide the heat of vaporization of the liquid	Little or no carrier gas is required to remove the vapors released from the solids
Heat transfer	Heat-transfer medium is in direct contact with the surface of the material to be dried	Heat needed to vaporize the solvent is transferred through a wall
Cross-contamination risk	Persists	Avoided, because the heat transfer medium does not contact the product
Solvent recovery	Difficult since there is a large volume of gas to be cooled to recover the solvent	Easier because of limited amount of non-condensable gas encountered
Dusting	High	Minimized because of small volume of vapors involved
Energy efficiency	Significant energy lost through exhaust gas	Higher energy efficiency because the energy lost through the exhaust gas is greatly reduced
Handling toxic materials	Not suitable	Suitable
Evaporation rate	Higher than conduction dryers	Drying rates are limited by heat transfer area, lower production rates
Cost	High	Higher initial cost; difficult to design, fabricate and maintain

Conduction (contact drying)

Contact drying involves an indirect method for removing liquid from a solid material by applying heat. In contact drying, the heat-transfer medium is separated from the material to be dried by a metal wall. Heat transfer to the product occurs predominantly by conduction through metal walls and impellers. Mixing is required to ensure contact between material surface and the heat-transfer surface. Heat-transfer fluids are often steam, hot water, or a heated oil [*1*].

Conduction drying can be particularly useful in cases where solvent needs to be removed from a solid material in a closed-cycle drying circuit, and can be a good choice for materials that are not heat-sensitive. Conduction drying could be an option when the solid material can tolerate heat-transfer-fluid temperatures well over the boiling point of the liquid.

An important benefit of conduction drying is its high thermal efficiency. Only a low flow of drying gas (air or nitrogen) is needed to sweep the evaporated liquid out of the dryer, and the air is not used as the heat source, as it is in convection drying. Smaller volumes of drying gas exiting the dryer means lower exhaust-gas enthalpy, and therefore, a higher thermal efficiency for the dryer.

Because of the higher thermal efficiency, conduction drying may offer economic and environmental advantages over convective drying approaches. Some of the possible cost benefits result from the smaller solvent recovery equipment required and the lower chilled-water requirements for the condensing operation.

Another important potential benefit for conduction drying is the minimized risk of cross-contamination. In conduction drying, the heat-transfer medium does not contact the product being dried.

Convection drying

Convection drying involves removing moisture from the surface of solid materials through contact with gases, usually forced air. Convective drying uses the sensible heat of the fluid that contacts the solid to provide the heat of vaporization of the liquid. Solid materials can be exposed to the heated gases via various methods, including the following:

- Gases can be blown across the surface (cross-circulation)
- Gases can be blown through a bed of solids (through-circulation; used when solids are stationary, such as wood, corn and others)
- Solids can be dropped through a slow-moving gas stream, as in a rotary dryer
- Gases can be blown through a bed of solids that fluidize the particles. In this case, the solids are moving, as in a fluidized-bed dryer
- Solids can enter a high-velocity hot-gas stream and can be conveyed pneumatically to a collector (flash dryer)

Dryer types that rely heavily on convection include fluidized-bed dryers, flash dryers, spray dryers and conveyor dryers. Fluidized-bed dryers and flash dryers are used in cases where particles have high surface-to-volume ratios. Spray dryers are typically associated with drying solids from liquids or slurries. Conveyor dryers are typically used with larger particles or friable materials because they can minimize particle breakage with a static material bed [*1*].

Equipment selection

Determining the best drying approach for an application and selecting specific drying equipment depends greatly on the physical characteristics of the material to be dried. Table 1 compares the two drying approaches discussed here according to a set of properties [2]. ■

References

1. Walsh, John and Whaley, M., Supplier's Tips, *Powder and Bulk Engineering*, April 2014.
2. Parikh, D., Solids Drying: Basics and Applications, *Chem. Eng.*, April 2014, pp. 42–45..

Polyacrylonitrile Production

By Intratec Solutions

Polyacrylonitrile (PAN) is a polymer resin mainly composed of acrylonitrile. This important acrylic resin is used in the production of a wide range of products, from ultra-filtration membranes to high-quality carbon fibers.

The process

The process discussed here comprises two main phases: first, acrylonitrile is polymerized with methyl acrylate comonomer and additives in an aqueous solution of demineralized water; then the copolymer is passed through transformation steps to become a fiber. Figure 1 presents a simplified flow diagram.

Polymerization. Initially, a mixture of acrylonitrile, methyl acrylate and itaconic acid is fed continuously to a continuous stirred-tank reactor (CSTR) where the polymerization takes place. The reaction is initiated by feeding aqueous solutions of potassium persulfate (oxidizer), sulfur dioxide (reducing agent), ferrous iron (promoter), and sodium bicarbonate (buffering agent). The heat of polymerization is removed by water circulated in a reactor jacket.

Next, an iron-chelating agent is added to the reactor overflow, where it acts as a chain stopper for the polymerization. The reactor overflow, an aqueous slurry of polymer particles, is fed to a monomer-separation column, in which unreacted monomer is steam-stripped from the slurry. The monomers recovered are returned to the reactor.

Pelletizing. The polymer is pelletized, dried, ground and then converted to fiber through subsequent steps involving physicochemical transformations, as briefly described below.

Wet spinning. The polymer is dissolved in dimethylformamide (DMF), forming a dope. The dope is pumped through a spinneret into a precipitating bath, for the coagulation of the solution thread line into a highly swollen gel yarn.

Washing, stretching and finish. The fiber is washed countercurrently by hot water, for the removal of the solvent. The fiber is then stretched while passing over rollers with adjustable speed. The stretching imparts fiber strength. Subsequently, silicone is applied as an aqueous emulsion to the fiber, to act as lubricant and antistatic agent.

Drying, crimping and relaxation. The fiber passes through drum dryers for water removal. Tows are crimped by means of a stuffer box crimper. Crimping provides interfilament cohesion across the tow and facilitates subsequent handling. Then the fiber is fed to an autoclave for a relaxation process that reduces the tendency for fibrillation and increases the fiber dimensional stability.

Production pathways

PAN production consists of the copolymerization of acrylonitrile — the main component — and other comonomers, in such a way that different manufacturing routes are related to different sources of such raw materials. In this context, typical PAN production routes are based on acrylonitrile manufacturing. Different pathways for polyacrylonitrile (using methyl acrylate comonomer) production are presented in Figure 2.

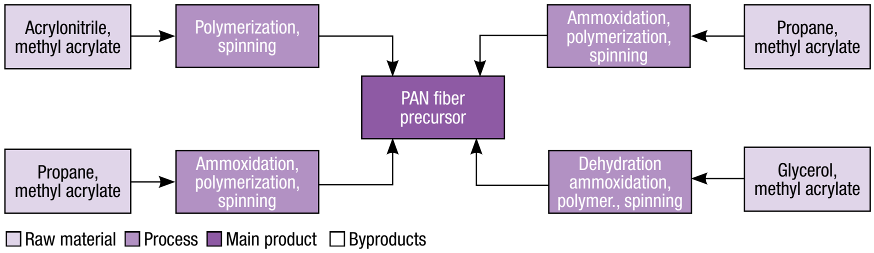


FIGURE 2. Multiple production pathways exist for polyacrylonitrile fiber

Economic performance

The total operating cost (raw materials, utilities, fixed costs and depreciation costs) estimated to produce polyacrylonitrile was about $4,600 per metric ton in the first quarter of 2015. The analysis was based on a plant constructed in the U.S. with capacity to produce 9,000 metric ton per year of polyacrylonitrile.

This column is based on "Polyacrylonitrile Production – Cost Analysis," a report published by Intratec. It can be found at: www.intratec.us/analysis/polyacrylonitrile-production-cost. ■

Edited by Scott Jenkins

Editor's note: The content for this column is supplied by Intratec Solutions LLC (Houston; www.intratec.us) and edited by *Chemical Engineering*. The analyses and models presented are prepared on the basis of publicly available and non-confidential information. The content represents the opinions of Intratec only. More information about the methodology for preparing analysis can be found, along with terms of use, at www.intratec.us/che.

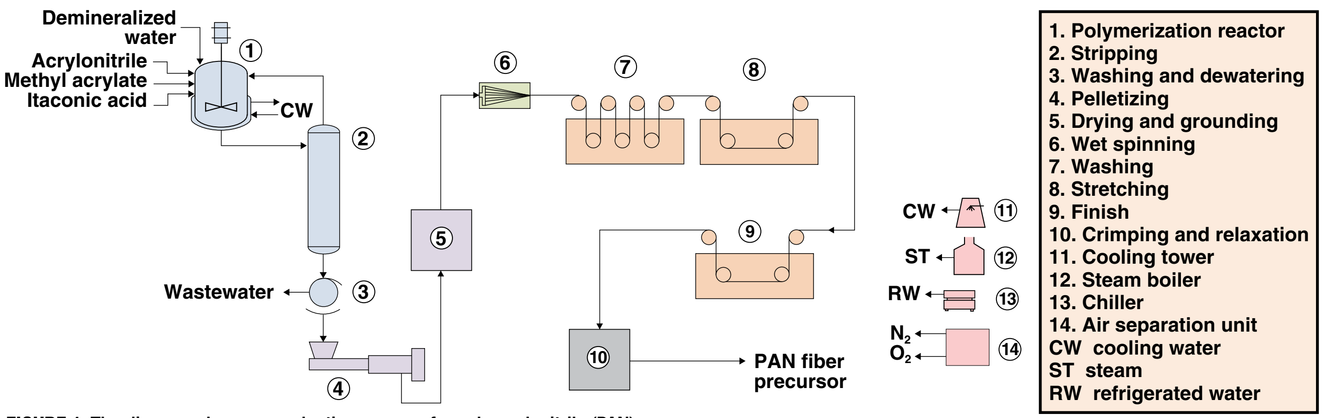


FIGURE 1. The diagram shows a production process for polyacrylonitrile (PAN)

Part 1

Protecting Instruments from Overpressure

Overpressure in a process can damage the seals in a pressure instrument's sensor or skew the zero point. Here's how to avoid the problem

Ehren Kiker
Endress+Hauser

IN BRIEF

PRESSURE MEASUREMENT

PROCESS UPSETS

MANIFOLD ISSUES

TOUGH INSTRUMENTS

EXPANDING FILL FLUIDS

CERAMICS

Pressure is one of the most commonly measured variables in the chemical process industries (CPI). Accuracy and, more importantly, stability are critical to maintaining a safe, reliable process and maximizing uptime. Modern pressure instruments are highly accurate and provide excellent long-term stability, with a variety of diagnostics to provide greater insight into the process, but they are still highly susceptible to overpressure events. By better understanding the application and the sources of overpressure, as well as knowing options available from manufacturers, process engineers can better avoid the possibility of costly process problems caused by overpressure.

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FIGURE 1. Overpressure can damage a pressure instrument's sensor, such as this differential pressure device

Pressure measurement

Pressure is typically measured by an instrument with a sensor and a transmitter. The sensor interfaces to the process to determine the pressure and sends this value to the transmitter. The transmitter converts the signal from the sensor to a type suitable for transmission to a control and monitoring system, such as 4–20 mA or a digital fieldbus-communications protocol.

Modern electronic pressure instruments (Figure 1) are safe and reliable, with onboard diagnostics and extremely sensitive sensors that provide insight into the process and flexibility in application. They can measure either differential pressure (that is, the difference between two pressures in a pipe or vessel) or static pressure (gage or absolute pressure).

In addition to the sensor, a transmitter consists of several important components. The sensor body is the machined component that houses the sensor. The sensor diaphragm(s) serves to keep process fluid from coming into direct contact with the sensor, while allowing the process pressure to be measured by the sensor. The system may also incorporate a manifold that allows the transmitter to be isolated from the process for removal or calibration without having to shut down the process.

In many cases, the complete measuring system also includes remote process seals and capillaries with hydraulic fill fluid designed to provide added protection to the transmitter in the case of excessive process temperatures, aggressive process media or process media that could plug impulse lines. While these measuring systems are highly reliable, there are many common issues that can affect pressure measurement and speed of response, such as errors caused by ambient or process temperature swings.

These issues can typically be addressed by correct selection and installation (for instance, proper fill fluid selection, insulating and heat tracing of capillaries, and so on) of the pressure instrument. But there is one common issue that is difficult to avoid but can have severe consequences not only for the sensor, but for the process as well: overpressure.

Overpressure occurs when excessive pressure is applied to one, or both in the

case of differential pressure, of the sensor diaphragms of the instrument. This can be either directly to the process diaphragm of the instrument, or to a diaphragm seal either directly connected to the instrument, or remotely connected via a capillary with a non-compressible fill fluid.

Typically, there are three main causes of overpressure in a process: process upset conditions, excessive cycling of valves and incorrect use of manifolds.

Process upsets

Process upset conditions occur when the pressure, temperature or both exceed the acceptable limits for the pressure instrument's sensor. This could occur during normal operation but is most likely to occur during startup or shutdown. The most obvious effect occurs when there is an actual overpressure applied to the sensor. The less obvious, but more likely cause, is when the transmitter experiences elevated ambient or process temperatures. This can cause the fill fluid in the seals and capillaries to expand, while also increasing the internal pressure of the fluid such that an overpressure is applied to the sensor.

Even when process conditions are within acceptable limits, there is the possibility of overpressure being applied due to cyclic pressures on the sensor. The repeated "on/off" cycling of pressure from a high pressure to a low pressure can have the same effect as overpressure, causing damage to both the diaphragm and the sensor.

Manifold issues

More common than process upset conditions is when manifold valves (Figure 2) are improperly closed and opened. Manifolds are used to isolate the instrument's sensors from the process for re-zeroing/recalibrating or removing the instrument without the need to shut down the process. Three-valve and five-valve manifolds are regularly used with differential pressure (DP) instruments on DP flow or DP level applications.

When closing the process manifold valves, it is important to make sure the valves are fully closed and seated before opening the equalizing valve. A common mistake is opening the equalizing valve before completely closing one or both process valves, allowing line pressure to leak past the valve and impact the sensor diaphragms. Even the most seasoned instrument technician has probably made this mistake on more than one occasion.

In the best-case scenario, an overpressure event has no discernible effect on the sensor's measurement. Most overpressure events, however, result in a shift of the zero-point of the sensor, resulting in the need to re-zero the instrument's transmitter. In some cases, the effect can be severe enough that the instrument must be recalibrated — most likely by removing it from the process and placing it on a bench calibrator.

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FIGURE 2. Five-valve manifold (to left of transmitter) is used to isolate a pressure instrument's sensor so it can be removed from the line, re-zeroed or calibrated

While neither of these types of incidents should require replacement of the instrument, if left unchecked they can have unforeseen impacts on process operations that could result in unplanned shutdowns. In addition, repeatedly having to re-zero or recalibrate a pressure instrument due to overpressure events uses manpower that could be employed elsewhere in the facility.

In situations where the overpressure event is severe enough or prolonged enough, the diaphragm or the sensor can be irreparably damaged. When this occurs, the only option is to replace the damaged instrument. It can be especially costly and time consuming if the instrument has remote diaphragm seals and capillaries.

Tough instruments

While the effects of overpressurizeation sound dire, it's incorrect to assume that all instruments are built the same way and have the same limitations when faced with an overpressure event. It's important to understand if the specific instrument has internal systems to minimize the effects of overpressure, or if the sensor itself can handle high overpressure

FIGURE 3. Remote seals, as on this pressure instrument, protect the sensors

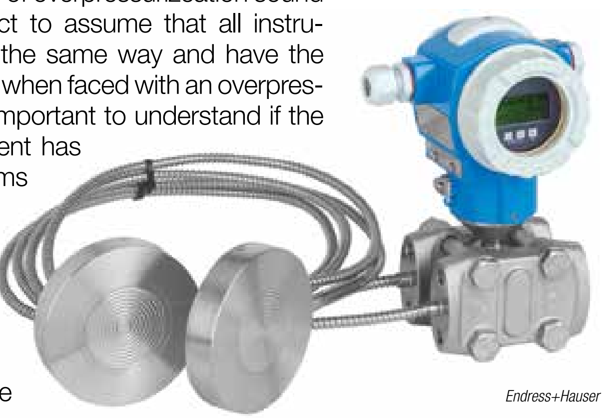

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FIGURE 4. This remote seal protects the fill fluid from high temperatures

without the possibility of hysteresis errors (the difference in output at any measurement point when approaching the point first with increasing and then with decreasing pressure) or a significant risk of failure.

To understand what steps can be taken by manufacturers, it's important to consider the two most common types of pressure instrument sensors on the market: strain-gage sensor typically used for differential pressure measurement and ceramic sensors typically used for static pressure measurement.

Strain-gage sensors come in a variety of types such as piezoresistive, resonant frequency or capacitance styles. Regardless of the sensor type, there are two things common to strain-gage instruments: metal process diaphragms and fill fluid.

Process diaphragms prevent direct contact of process fluid with the sensor. This can be accomplished both with either internal process diaphragms or remote seal diaphragms (Figure 3), each of which can also protect the entire instrument from elevated temperatures or aggressive chemicals.

These measuring diaphragms can be made from a variety of metals, but all are extremely thin to allow for greater sensitivity to small changes in pressure. As a result, these diaphragms can easily be damaged or even ruptured due to overpressure. In addition, overpressure or repeated cycling of process pressure can cause the diaphragm to deform, resulting in a shift in the measurement. It's important to remember that this can happen even at relatively low overpressures due to repeated cycling of pressure, even within what may be considered acceptable limits.

FIGURE 5. This ceramic sensor can withstand three times the overpressure of an oil-filled sensor

Expanding fill fluids

Hydraulic fill fluids are non-compressible and are used to transfer the pressure from the process diaphragm to the instrument's sensor. There are a wide variety of fill fluids that can be used for different applications. The most common types are silicone oils, which come in grades to handle different process temperatures.

In addition, there are inert oils for applications where silicone is not acceptable, and food-grade fluids for use in food and beverage or pharmaceutical applications. All fill fluids can expand during upset conditions, such as elevated process or ambient temperatures or pressures.

This expansion can cause significant overpressures on both the process diaphragm and the sensor, which can result in measurement shifts or irreparable damage to the diaphragm or sensor.

There are several ways for manufacturers to design oil-filled pressure instruments to better withstand overpressure events. These include taking steps to minimize the amount of fill fluid in the instrument, and then designing mechanical systems to absorb the force of the overpressure and keep it from reaching the instrument's sensor.

As previously mentioned, fill fluid expands and contracts according to the process and ambient temperatures of the application. The best way, therefore, to minimize the expansion effects of the fill fluid is to use as little fill fluid as possible. This requires careful design of the instrument to minimize the volume of fill fluid while still having enough fill fluid to maintain the sensitivity of the measurement.

One way this can be accomplished is by minimizing the space between the process diaphragm and the sensor body. Many manufacturers machine the sensor body to match the pattern pressed into the diaphragm. Some even go the added step of hydroforming the process diaphragm against the machined sensor body to ensure an exact match between the diaphragm and the sensor body, resulting in the smallest possible volume of fill

fluid in the system.

Some manufacturers are looking to new diaphragm designs on remote seals to better address the effect that temperature has on the fill fluid and the subsequent overpressure that can occur during temperature swings. Remote seals such as the one shown in Figure 4 are designed to greatly reduce the temperature-induced error caused by fill-fluid expansion and the resulting overpressure applied to the sensor.

Manufacturers are also implementing internal systems to mechanically minimize the force of overpressure reaching the sensor, allowing the sensor to withstand overpressure without the damage or shift to measurement output normally associated with such events. To address overpressure, these manufacturers are including internal overpressure diaphragms in the sensor body. Rather than act as a thin, sensitive metal barrier to transmit the pressure to the sensor, these overpressure diaphragms are thicker pieces of metal designed to absorb the force of the overpressure.

One of the easiest ways to eliminate fill-fluid problems is to employ electronic DP level systems, which eliminate the need for impulse lines or capillary tubes because the sensors connect to the transmitter via wiring. Each sensor is equipped with electronics to convert its reading to a value suitable for sending to the transmitter over commonly used electrical wiring.

Ceramics

Oil-filled transmitters make up the majority of industrial installations of pressure instruments, but there are many static pressure applications that can use ceramic sensors that are particularly well-suited to handle overpressure. The major difference between ceramic sensors and oil-filled sensors is the use of a ceramic diaphragm versus a metal diaphragm. The other is that ceramic sensors do not have fill fluid.

The use of ceramic (Figure 5) as the diaphragm material has several benefits in terms of overpressure. The first is that ceramic is extremely hard and rigid, so it will not deform as easily during an overpressure condition. The second is that ceramic is hysteresis-free, meaning that it does not develop a memory when excessive pressure is applied. As a result, a ceramic sensor can be exposed to up to three times the amount of overpressure of an oil-filled sensor — with no adverse effect on the measurement, no memory developing at the diaphragm and no damage to the diaphragm or sensor. ■

Edited by Gerald Ondrey

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Part 2

Prevent Product Release with Pressure Protection Systems

Closing off pressure upstream can minimize the need for pressure-relief systems to open, avoiding product waste and possible environmental damage

Erik Mathiason
and
Afton Coleman
Emerson Automation Solutions

IN BRIEF

RELIEVING PRESSURE AT THE SOURCE

LOOKING AT THE INDIVIDUAL PARTS

IMPLEMENTING AN IMPROVED OVERPRESSURE PROTECTIVE SYSTEM

PUTTING IT ALL TOGETHER

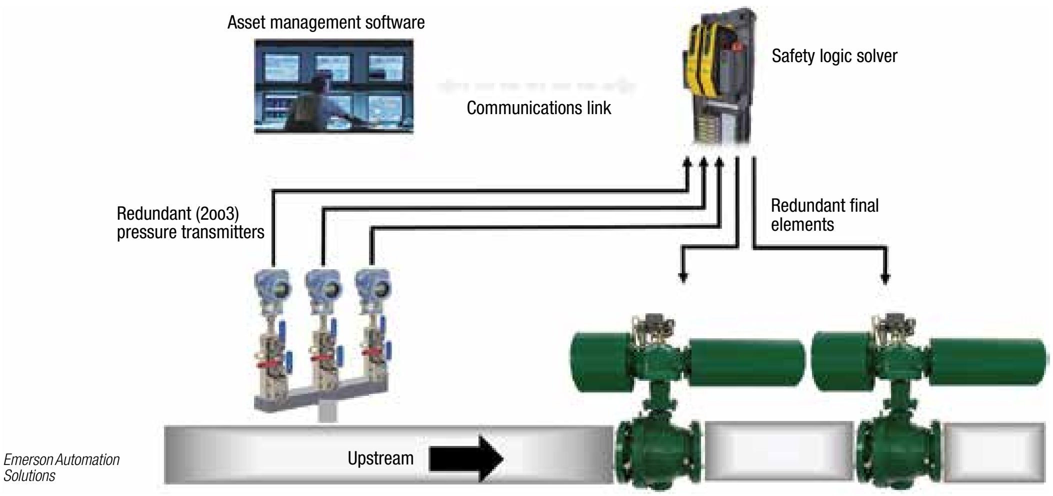

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FIGURE 1. The OPS is installed on a main line supplying a process unit. If the feed pressure passes a critical threshold, it shuts off the feed prior to the PRV opening

Any system within a chemical process industries (CPI) manufacturing plant that is pressurized, or could experience pressure during an upset or malfunction, must have a pressure-relief mechanism. This helps alleviate a situation where pressure capable of bursting vessels or piping develops. The first approach for protection is a mechanical device designed to open at a specific pressure to release the contents. Typical examples are pressure-relief valves (PRVs) or rupture disks. These open and allow the contents to escape to the atmosphere, a flare manifold or a collection system.

The problem with this approach is the amount of product lost, along with the potential for an environmental incident if the contents are not fully captured and treated appropriately. For example, a plant's flare system must have the capacity to handle large releases, which could require a more robust system than is needed on a routine basis. In other words, an overpressure incident involving product release through a mechanical pressure-relief system is expensive not only in lost product and potential environmental incidents, but also in the equipment necessary to handle the release. This can be made all the worse by a large process supply, such as that found in a pipeline. Without being shut off, the flow out of an opened PRV from a pipeline could be virtually inexhaustible.

In some situations, it may be more practical to change the approach from releasing the pressure to cutting it off upstream at the source as quickly as possible to minimize or prevent product release through a PRV. Although this seems like an obvious solution, it happens less than one would expect; product is still lost with all the associated costs and inconveniences. It is

important to consider alternate solutions to address safety and production challenges.

Relieving at the source

As a typical example, an overpressure protection system (OPS) (Figure 1) can be used to protect a reactor by shutting off the pressure source where it reaches the critical vessel. One approach shuts down the system when it reaches 116% of the rated pressure, and before the PRV is set to open. This is a basic safety instrumented function (SIF) and should be part of the larger safety instrumented system (SIS) protecting the process unit or larger plant.

If designed and applied properly, the OPS may be able to mitigate the need for an additional pressure-relief system. This is particularly critical when the pressures involved make a PRV impractical, or the systems designed to handle a product release might be inadequate for the potential volume. Such situations can emerge when a plant is changed to increase production or a process unit is modified. The capital expenditure costs of enlarging a flare system can be much higher than adding an OPS.

There are situations where an OPS cannot be used to replace a conventional pressure-relief system, such as compressed air or steam. In such situations, it becomes necessary to have the two systems work together, with the PRV serving as a backup.

The design and implementation of an OPS must be approached just like any other SIF following the procedures outlined under the International Electrotechnical Commission's (IEC) 61511 standard (ISA-84). If this standard is not familiar, there are many resources available covering the topic in far greater depth. IEC 61511 provides a complete safety lifecycle approach, covering everything from conceptual process design with hazard analysis and risk assessment, all the way to operation, evaluation and management of change. Any OPS should be evaluated and designed by experts with competency in safety standards and process design within this framework. For purposes of this discussion, we will concentrate on the middle steps of the process covering detailed design and installation, but all the other elements need to be included.

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FIGURE 2. Safety-certified smart pressure transmitters have internal capabilities to perform continuous loop diagnostics to spot problems such as clogged impulse lines

Looking at the individual parts

Returning to the earlier example, let's focus on the OPS function itself. It is a basic SIF designed to shut down flow in the event of an excessive pressure incident. Since it is a SIF, it has three basic parts: a sensor, a logic solver and a final element. These can be simple or highly sophisticated, but any components used must be certified to operate in a SIS context, with any uncertified components justified as proven-in-use.

A SIF must be able to perform its function independently without depending on any larger automation system, or even plant utilities. Should there be larger failures in the unit, it must be able to shut off the flow at least once and remain closed. A bare-bones system could use a pressure switch connected to a simple logic solver and a shut-off valve designed to fail closed in the event of a loss of electric power or compressed air. This can probably do the job, but will be difficult to test,

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FIGURE 3. This 12-in. valve is typical of a shut-down valve for OPS service. It is a trunnion-mounted ball valve assembly with a scotch yoke pneumatic spring return actuator and digital valve controller

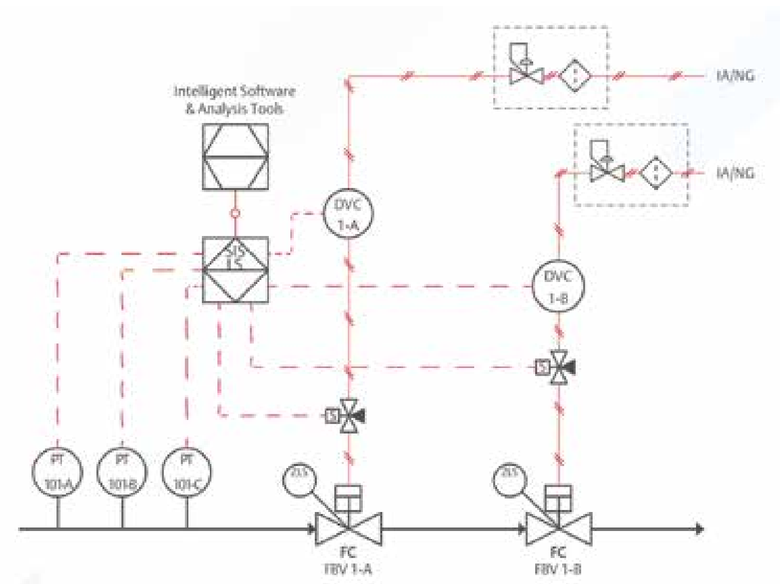
will not provide any diagnostic information, and won't have any capability to minimize false trips. One shutdown caused by a false trip or pressure switch failure will likely cost more than installing a more sophisticated system.

Implementing an improved OPS

The same sophisticated technologies that make instrumentation smart and control systems more effective can also apply to SIFs. Let's look at how they might be applied in this situation.

The sensor can be a pressure transmitter (Figure 2), which is safety-certified and able to provide diagnostic information in addition to a basic pressure measurement. A pressure transmitter with advanced capabilities can evaluate the complete measurement system, extending diagnostics to capture over-pressure scenarios and improve reaction time. Speed is critical because the OPS must respond immediately to initiate the pressure protection system and protect the downstream equipment.

FIGURE 4. This typical OPS monitors the feed to a reactor or other part of a process unit for high pressure



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Advanced transmitter capabilities can include functions such as plugged impulse-line detection, which can be communicated to the logic solver via a wired or wireless digital communications protocol. This valuable information can be passed on to the larger process-control and maintenance systems without interfering with the basic safety function.

While one transmitter may be sufficient to provide the safety function, highly critical systems where shutdowns are exceptionally costly may call for three pressure transmitters arranged in a two-out-of-three (2oo3) voting scheme to reduce the potential for a false alarm caused by a transmitter malfunction. Using this approach, at least two transmitters must report an overpressure incident to trip the system and cause a valve closure. This also provides a backup if one or even two transmitters are damaged or malfunction.

We'll come back to the logic solver in a moment, but let's first look at the final element. The ultimate purpose of an OPS is to close a valve to cut off the source of pressure. It may be a single valve, but as the diagram shows, it may also be two valves in series so that either one can close and isolate the process. Having two valves eliminates a valve malfunction as a single point of failure.

Valves in this service should be safety-certified, and must fail closed (Figure 3). If there is a loss of any or all plant utilities, such as compressed air to a valve, or if communication is cut off to the logic solver, the valve must close itself and stay shut. To handle these and other similar situations, fail-closed valves are typically spring loaded and have to be held open by the instrument supply medium, such as compressed air. But as mechanical devices, a potential failure mode is becoming stuck in one position, leaving them unable to do their job when called upon.

This is particularly important for SIFs, since these valves don't normally change position very often. Fortunately, the actuators that move these valves can include a smart digital valve controller (DVC). It can perform self-diagnostics and determine if corrosion or some other condition is causing a valve to require more force to move and can detect when there is a change in the compressed air supply, power quality or whatever other motive force they may depend upon. The DVC performs operations that can assist

with proof testing as well as partial-stroke tests, gathering and evaluating performance data. Proof and partial-stroke tests are important periodic tests that confirm operability in accordance with IEC 61511. Data gathered can be passed to the logic solver as well as asset management tools, which can also be utilized by operations and maintenance personnel to make predictive maintenance decisions.

Depending on the nature of the process, the valves used with an OPS can be quite large, yet they must close quickly and positively in a controlled manner. Large actuators sometimes need to be powered by hydraulic systems to have sufficient force to close. Even large valves must have actuators including stored energy systems, enabling them to fail closed. If driven by stored hydraulic pressure, this pressure must be monitored to ensure it is available when called upon.

The logic solver ties together the OPS. In the most basic form, it might be little more than a glorified relay, responding to a binary switching signal from a digital sensor and telling its valve to close, but it can also provide insight into the safety function, and even the process, if provided with the sophisticated functionality available today.

A smart logic solver has the capability to work with three pressure transmitters in a 2oo3 voting scheme, and it can tell if a transmitter is malfunctioning by reading the diagnostic information. It monitors all functions continuously, and if there is a problem, it can compensate while warning operators of the situation. The same applies to valve actuators, since they can receive, interpret and act upon diagnostic information from smart transmitters and actuators.

Putting it all together

A typical system could use three pressure transmitters, each sending measurement and diagnostic data to a logic solver (Figure 4). Using a 2oo3 voting scheme, the system will trip if line pressure exceeds the setpoint and it is detected by at least two of the transmitters. The shutoff valves close by spring actuation and are held open by a regulated compressed-air supply.

If the system trips or there is a larger failure in the unit, the solenoid valve or digital valve controller cuts off the compressed-air supply and dumps it from the actuators, causing the valves to close. The DVCs monitor air-line pressure and valve function. They can also perform partial-stroke valve tests automatically to determine if the valve condition is changing. All of this is monitored by the logic solver using analysis tools, which can warn operators and maintenance personnel if there are any changes in equipment or performance.

Creating an effective OPS requires selecting and integrating the right components following analysis in accordance with IEC 61511. Once designed, it must be evaluated as a system, usually by a third-party, to ensure it can perform as expected under the appropriate SIL requirements. It can be installed with less process downtime compared to the cost and complexity of a flare system expansion project. An OPS provides a variety of cost benefits, including the following:

- Reduces or eliminates the need for resizing the flare or relief manifold
- Reduced process downtime
- Reduced number of flaring events and volume released
- Reduced manual system testing

As environmental regulations continue to become more stringent and the ability to keep qualified operators grows more difficult, these systems will be increasingly considered for both operational and financial reasons. ■

Edited by Gerald Ondrey

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Field Performance Testing for Centrifugal Compressors

Determining the field performance of centrifugal compressors can benefit operation and maintenance of the equipment. Presented here is an overview of the parameters that must be measured and calculated for effective testing

Nathan Poerner, Tim Allison and Hector Delgado
Southwest Research Institute

IN BRIEF

Compressors are used in all areas of production and in many different sectors of the chemical process industries (CPI). Production facilities can have a number of compressors of different types and in a range of sizes. The three main compressor types are centrifugal, axial and reciprocating. This article focuses on centrifugal compressors (Figure 1) and how field performance testing can be carried out in this equipment class to benefit the end-users, manufacturers and maintenance personnel. The benefits of field testing for existing units include the ability to quantify performance deterioration as a function of equipment age, to determine if and when certain maintenance tasks should be performed, and to identify specific equipment components for product improvement. For new units, field testing helps validate product performance.

FIGURE 1. Performance tests on a multi-stage centrifugal compressor, like the one shown here, will quantify the factors of energy, head, work and flow

Performance principles

Regardless of compressor type, there are certain terms and parameters that will be used to define and quantify equipment performance. Primary among these are energy, head, work and flow. Energy is a parameter of the fluid in question, and will most commonly be defined as the total enthalpy of the fluid. Enthalpy is a combination of the kinetic (fluid velocity) and potential (static pressure) energy of the fluid. Because enthalpy is a derived term, there is no direct way to measure the enthalpy of a fluid. Instead, it is common practice to rely on equations of state (EOS) that relate measurable parameters (most commonly temperature and pressure) to derived terms, such as enthalpy and entropy, for a given fluid composition. A variety of EOS are available, and each will be best applicable for only a given set of fluid compositions over a specific range of operating conditions. Because of this, it is important that all parties involved in a performance test understand and accept the chosen EOS.

Head is a measure of the increase of energy within a fluid, and is typically defined as the change in enthalpy of the fluid between the suction and discharge conditions. Work is the mechanical means of transferring energy into the fluid.

Flow can be defined in terms of mass or volume, with volume taking one of two common forms. The first is the capacity, or actual flow, which considers the volume flow for the actual pressure and temperature of the fluid, usually at inlet conditions. The second

is referred to as standard, or normal, flow, and calculates the volume flow for the given mass flow at a "standard" condition. For example, this could be a temperature of 60°F and pressure equal to 14.7 psia. For either volume flow term, the mass flow would be identical.

Measurements and calculations

The data gathered to determine performance include temperatures, pressures, flow and gas composition. Temperatures and pressures are relatively straightforward to collect using a variety of available sensors. However, flow can be a bit more complex to determine. Such measurements often rely on additional measurements, but there are still a number of sensor options available. Gas composition is probably the least accessible data to obtain, either requiring an online gas chromatograph (rarely available in most installations), or sampling of the fluid for analysis by a laboratory after the testing.

In addition to these base measurements, some common performance parameters that will be generated include the work, head and efficiency. Work and head, discussed earlier, lead to the definition of efficiency, which can be defined in many different ways.

First, there is the thermodynamic efficiency that only involves the actual compression process, and relates the actual head to an ideal head. The ideal head can also be defined in multiple ways; the two most common are isentropic and polytropic. It is critical to understand which of these two definitions is being used in a given case. Isentropic efficiency calculations assume that the thermodynamic process is adiabatic, and compares the actual head to the ideal head, with entropy at the suction and discharge assumed to be the same.

Polytropic efficiency compares the actual head to the head conceptually calculated by breaking the entire compression step into a number of smaller steps, with some heat added at each step. The two processes converge at low pressure ratios, yielding identical results. One benefit of using polytropic efficiency is that for multistage machines, if all stages have the same polytropic efficiency, then the entire machine polytropic efficiency is equal to the stage polytropic efficiency. Overall machine isentropic efficiency in this case will be lower than the stage isentropic efficiency.

Another way that efficiency can be defined is the mechanical efficiency of the compressor. This usually involves additional measurements to evaluate the mechanical work going into the compressor, usually through the use of a torque-meter. This allows calculation of the compressor efficiency as the actual head compared to the mechanical work going into the compressor.

Finally, a total system efficiency can be generated if measurements are taken of the total energy being put into the system. For electric-motor-driven compressors, the total energy can be determined based on electrical measurements and knowledge of the motor. For engine- and gas-tur-

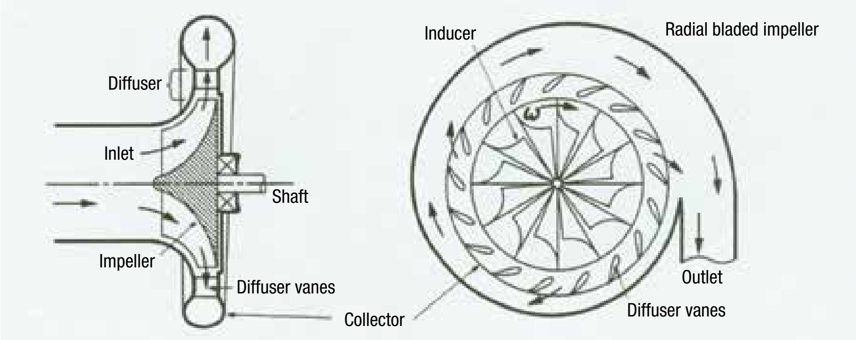


FIGURE 2. Centrifugal compressors have a rotating impeller that imparts head to a fluid

TABLE 1. NON-DIMENSIONAL PERFORMANCE PARAMETERS

Specific volume ratio	$v_S / v_D = \rho_D/\rho_S$
Flow coefficient	$\phi = Q_s / (ND^3)$
Machine Mach Number	$Mm = U / a_s$
Machine Reynolds Number	$Rem = Ub / \upsilon_s$
Head coefficient	$\psi = H / U^2$
Work input coefficient	$\mu = \psi / \eta$

$\rho_{(S,D)}$ – Fluid density at compressor suction, discharge, kg/m^3
Q_S – Volumetric flowrate at compressor suction, m^3/s
N – Rotational speed, rad/s
D – First stage impeller/rotor tip diameter, m
U – First stage rotor tip speed, m/s
a_S – Fluid speed of sound at compressor suction, m/s
υ_S – Fluid kinematic viscosity at compressor suction, m^2/s
b – Characteristic length: passage exit width of first stage impeller for centrifugal compressors, chord length at tip of first stage rotor blade for axial compressors, m
H – Head (can be isentropic or polytropic), J/kg

bine-driven compressors, knowing the fuel composition and flowrate can be used to determine total energy input.

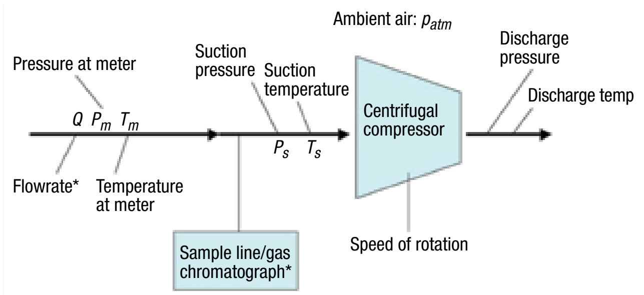
Centrifugal compressors

A centrifugal compressor uses the rotating energy of an impeller to impart head to a fluid primarily by first accelerating the fluid, and then converting the kinetic energy into potential energy by slowing the fluid. The typical internals of a centrifugal compressor are shown in Figure 2. Fluid enters the compressor and is directed in the inlet toward the rotating impeller. The blades on the impeller are designed to accelerate the fluid either axially or radially. There can be a potential energy rise due to the impeller blade geometry, but the majority of energy increase after exiting the impeller will be primarily kinetic. In order to convert this energy into a static pressure, the diffuser section will convert the kinetic energy into static energy by slowing down the fluid flow before it is directed to the outlet in the collector.

The design of each of these individual components can have a significant effect on the compressor performance, and therefore, must be considered carefully for each specific application. In addition, a centrifugal compressor can use multiple impellers in many different configurations to either increase the operating flow or compression ratio of the whole unit, with each individual impeller and diffuser sets of the compressor possibly comprising different designs.

Field performance testing

One of the first things to consider when planning a performance test of a centrifugal compressor is the placement



*Flowrate measurement and gas sample may be on suction or discharge side. Suction side is recommended.

FIGURE 3. The diagram shows the locations of test instrumentation for a centrifugal compressor [*4*]

of sensors for gathering the operating conditions of the system. The ideal layout of measurements is shown in Figure 3. Included is a flow-measurement device (preferably on the suction side of the unit), instruments for measuring temperature and pressure on both the suction and discharge sides (although discharge temperature is not necessary), a means for determining gas composition, a means of measuring the unit rotational speed, and instruments to measure atmospheric conditions (mostly to correct gage-pressure measurements to absolute). Specific locations of the actual sensors should be selected to avoid errors due to flow distortion from piping, valves or upstream sensors. Acceptable locations for sensors are defined in industry standard documents available in the references section.

From these measurements, and with the aid of an appropriately selected and agreed upon EOS, performance parameters of the compressor unit can be generated, including power, which is shown in Equation (1).

$$P_{compressor} = \dot{m}_{compressor}\left[h(p_D, T_D) - h(p_S, T_S)\right] \quad (1)$$

Assuming isentropic compression, the efficiency of the unit can be calculated according to Equation (2).

$$\eta_{compressor} = (\text{isentropic head / actual head}) = \frac{h_{is}(p_D, s_S) - h(p_S, T_S)}{h(p_D, T_D) - h(p_S, T_S)} \quad (2)$$

Where:
$s_S = s(p_S, T_S)$
$h(p,T)$ = enthalpy (heat content) of gas
$h_{is}(p_D, s_S) - h(p_S, T_S)$ = isentropic enthalpy change of gas
$s(p,T)$ = entropy of gas (a thermodynamic quantity representating unvailable energy; Necessarily increases with turbomachinery process due to frictional losses
$\dot{m}_{compressor}$ = compressor mass flowrate

Along with selecting the specific measurement sensors and determining their placement, preparation for the field performance test should also include the following items:

- *Defining the operating conditions to be used for the test points*
- *Selecting the approach for data reduction and test uncertainty.* The accuracy of calculated compressor performance depends on measurement uncertainty in the inlet and exit pressures and temperatures, gas composition, and mass flowrate. Uncertainties in performance can range from less than 0.5% in laboratory tests [*6*] to 2–3% or more in field measurements [*7*]. Overall measurement uncertainty depends on a large number of factors, including sensor accuracy and calibration, proper probe installation, signal conditioning, flow non-uniformity or unsteadiness, and heat loss from the system.
- *Specifying the ultimate objective of the test.* For example, in the case of an acceptance test, the field performance test results will be used as an acceptance criterion. In cases where the performance test data will be used for maintenance scheduling, engineers should specify how the data will be compared to previous measurements for equipment aging practices.

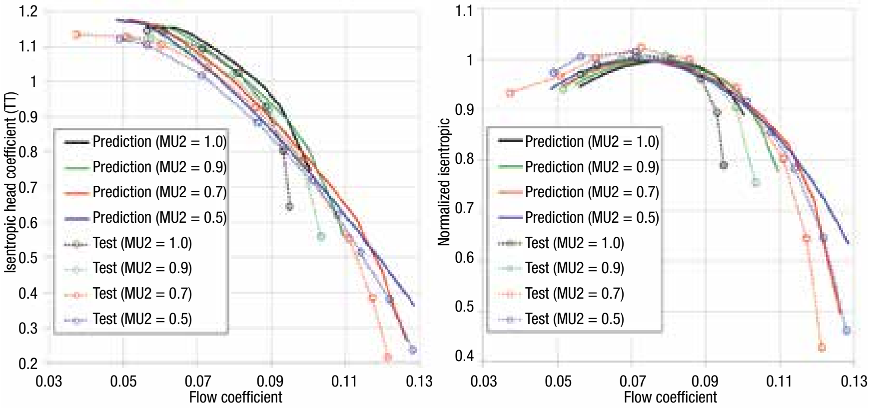


FIGURE 4. The graphs show example performance data for a centrifugal compressor [*5*]

When making a comparison between current and previous field measurements, it is important to recognize that measurements taken at different operating conditions cannot be directly compared. Therefore, it is necessary to either ensure that exactly the same operating conditions are duplicated for the two sets of measurements, or that there are non-dimensional parameters that can be used for comparing the data from tests taken under different operating conditions.

The six most common non-dimensional performance parameters used for centrifugal compressors are the specific volume ratio, Machine Mach and Reynolds numbers, and the head, flow and work input coefficients. These terms are defined in Table 1. Test conditions that closely match the specific volume ratio and flow coefficient are expected to produce similar results for the head coefficient and work input coefficient, with some variation due to different Mach and Reynolds numbers. These variations can be minimal or can be corrected based on test standards if the Mach and Reynolds number deviations are within allowable limits.

Example plots showing measured isentropic head coefficient efficiency versus flow coefficient are provided in Figure 4 for a single centrifugal compressor stage that was tested at multiple Machine Mach numbers (MU2). The predicted curves (solid lines) compare reasonably well with the measured curves (dashed lines), although the peak head and choke flow measurements are both below predicted values, and efficiencies near the design flow coefficient of 0.08 exceed predictions. The results for head coefficient show a typical characteristic of increasing head rise at lower flowrates, with a zero slope at low flow (surge point). The choke line at high flow is shown to be a strong function of the Machine Mach number. The normalized efficiency results show peak efficiencies near the design flow coefficient regardless of Machine Mach number, and also highlight reduced efficiencies and lower choke flow at high Mach numbers.

Concluding remarks

Field-performance tests can benefit compressors in all stages of equipment life. For new units, performance tests can validate product performance, while for existing units, performance tests can help to monitor for maintenance practices. In addition, performance can be used to determine an end-of-life metric.

Centrifugal compressor performance metrics are based primarily on measured temperatures, pressures, flows and gas compositions, as well as an appropriately selected equation of state. Careful selection and placement of measurement transducers are necessary to reduce test uncertainty. Proper planning of the testing is also necessary to define operating conditions and the desired ultimate objective of the test. ■

Edited by Scott Jenkins

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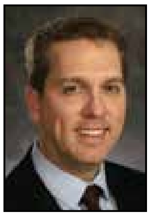

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Key Considerations for the Use of Portable Gas Detectors

Recent advances in gas-monitoring technologies can greatly increase worker connectivity and safety

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Honeywell Safety and Productivity Solutions

With increased automation, expanding areas of operation and smaller teams of field workers, monitoring personnel and ensuring lone worker safety are serious challenges for the chemical process industries (CPI). When operators and technicians are in danger, it is crucial to know their location and react in a fast and effective manner.

Recent developments in portable gas detection leverage new technology for improved protection of the industrial workforce by connecting wearable detectors with the distributed control system (DCS) through the cloud to provide expanded visibility of plant floor operations — helping to keep workers safe and promoting better emergency response (Figure 1).

FIGURE 1. Gas detection has always been essential in CPI operations, but as manufacturing complexes have expanded and operations teams have shrunk, remote monitoring technologies are increasingly required to ensure safety

Today's safety challenges

Process operations, such as those found in the CPI, often involve the use or manufacture of hazardous, flammable or toxic gases, which have the potential to escape and threaten those working within plants or possibly even those living nearby. Incidents may escalate, resulting in environmental damage, explosions or loss of life.

Plant workers have been carrying different types of portable gas detectors for years — one of them being their nose (Figure 2). So, it is already understood that detecting and avoiding noxious gases is a valuable capability. But some hazardous gases cause "olfactory fatigue," meaning that the exposed person's sense of smell diminishes over time.

Hydrogen sulfide (H_2S) is a particularly hazardous gas commonly found at industrial sites. It has a distinctive "rotten egg" smell at low concentrations. H_2S is relatively easy to detect at 0.01 parts per million (ppm), but above 100 ppm, it is no longer discernible. Olfactory fatigue can happen so fast that people can easily be overcome without realizing they are being exposed.

Low-oxygen areas are another significant threat to plant personnel. Human beings normally breathe air that is 20.9 vol. % oxygen under normal atmospheric pressure conditions. When the concentration of oxygen decreases even slightly, they immediately begin to feel the effects.

Tragically, there have been situations where a "lone worker" in an industrial facility succumbed to either a lack of oxygen or the presence of a toxic gas. By the time anyone became aware that the worker was in danger, it was too late.

Progress in gas monitoring

Portable gas monitoring was introduced over 50 years ago and has been evolving ever since. It is regularly used to detect toxic or combustible gas — or preferably to confirm the absence of such gases. Monitoring systems provide early warning of an abnormal situation before it becomes a significant threat.

Current technology has allowed the capabilities of portable gas-monitoring devices to be continuously extended. In the past, two detectors were needed to confirm there were no flammables, and the oxygen concentration was normal, before allowing entry into a confined plant space. Subsequent developments have allowed a single monitor to be set up with a pump and sampler to provide continuous indication with an alarm.

However, workers in many jobs face risks that require detectors that can handle multiple gases at once. These more complex multi-gas devices are also increasingly mandated by new regulations. A single

detector measuring four or more gases can be worn by each person entering a confined space, giving them an audible alarm signal if there is a gas hazard right where they are standing. Furthermore, the price of multi-gas detectors has come down so far in recent years that many companies provide a personal detector to every worker in the plant.

Advancement of portable devices

Experience has shown that gas detection is a critical requirement in the CPI and many other industrial sectors. When it comes to portable gas detectors, device adaptability is a key factor. Since hazardous gases can present themselves in a myriad of environments — spanning different industries — detection solutions need to be able to identify a variety of threats.

Unfortunately, traditional gas detectors and lone-worker safety monitors function like smoke detectors, alerting only the wearer and those in earshot of a dangerous environment or incident, potentially leaving workers alone for hours without help. Furthermore, traditional employee-worn gas detectors are normally autonomous solutions, meaning safety personnel have no realtime awareness of what is happening with the device and the worker it is meant to protect.

In the control and instrumentation industry, research and development teams have been working on advanced detection technologies that are leading to the introduction of more connected gas-detection systems. Advanced wearable gas detectors have become a viable solution for alerting personnel to hazardous vapors in the workplace. They typically are designed to monitor H_2S, carbon monoxide (CO), oxygen (O_2) and combustible lower explosive level (LEL) gases in a standard configuration, and can also be equipped to handle LEL, O_2, CO and hydrogen cyanide (HCN), making them suitable for a wider range of applications. Connecting safety managers with field workers at all times using wirelessly connected devices greatly enhances visibility of worker hazards, status and location. Many of these newer devices may integrate not only gas-detection capabilities, but also decrease the risks to lone or remote workers by providing additional information, such as:

- Continuous worker location monitoring
- Movement detection for automatic man-down alerts to prompt automatic check and immediate notification when a worker is down
- Indication of compliance status for each gas detector
- A view of the site map in the event of critical alarms, showing the location and exposure status of personnel in the field

FIGURE 2. Workers in industrial facilities cannot always depend on their noses to detect unsafe gas situations, especially in remote areas where they might be working alone for long periods of time

Increased connectivity

When it comes to industrial workers — especially those who do their jobs alone or in remote locations — the Industrial Internet of Things (IIoT) can do more than just get them better connected. Connected gas-detection solutions can enable plant- and enterprise-wide monitoring that harnesses the IIoT to provide realtime safety insights on gas threats, concentration, location, man-down and more.

One of the major trends in plant operation has been the evolution of smartphones and other handheld devices from potential distractions to effective productivity and safety tools. Some wireless multi-gas monitors can be paired with ruggedized, intrinsically safe smartphones to transmit gas readings over a cellular or satellite network (Figure 3). The phone can act as a personal communication hub for a worker, and can collect readings and alarms from, and send the data to, a remote system. Mobile devices and applications can also transmit worker information and location to the remote system, allowing safety managers to

FIGURE 3. Some gas-detection solutions feature connectivity with smartphones or tablets to transmit data over a satellite or cellular network

quickly react and provide necessary assistance in case of an emergency. The ability of safety managers in remote locations to access realtime safety and location data from a worker's wireless gas detector decreases safety risks for offsite or lone workers and can help to expedite required intervention if an incident does occur.

Other practical applications extend to carrying out a man-down rescue or plant egress with greater confidence and better outcome, or identifying an equipment malfunction in its early stages so that costly downtime can potentially be averted.

This safety intelligence enables users to do the following: immediately determine the location and severity of a gas alarm; get instant awareness of a worker in distress; make better decisions about rescue and evacuation; and proactively monitor their plant's safety, compliance and productivity.

Furthermore, the location and gas status of workers, whether they are in a confined space during planned maintenance or walking throughout a process unit during normal operations, are always being monitored. Safety personnel are notified of any threats, while giving team members assurance that someone always has their back.

Lastly, as with any IIoT technology, robust cybersecurity technology can ensure that data remains protected from increasing external threats. Secure remote connections to networks employed for gas detectors require a solid defense-in-depth strategy. Plant operators must also have a proactive strategy for managing firewalls, intrusion detection and a host of other items.

An integrated approach

Portable gas-detection systems can now be securely integrated with the plant DCS via the cloud to help safeguard workers and speed up emergency response. Data from wearable gas detectors play a key role in revealing hazards, enabling proactive steps to improve safety and assist with learning from incidents.

With this integrated approach, for instance, control-room operators can identify and stop trace gas leaks, flag non-compliant or malfunctioning gas monitors for removal from the field and more quickly generate safety reports for regulatory compliance, as well as help reduce maintenance costs. In addition, safety teams can take advantage of powerful tools embedded in the DCS to provide detailed trending, reporting and data analysis of the gas detectors to further ensure safe operations.

By unifying connected wearable gas detectors with their DCS, and thus enabling two-way communications between field workers and operations and safety personnel, CPI engineers can gain effective safety monitoring from the control room console. This includes access to critical alarm reporting from wearable gas detectors for hazardous gases and radiation exposure. This allows operators to enhance their situational awareness and leverage control-room resources for faster initiation and support of emergency responses. More robust detection solutions also serve to eliminate extra load in the control room under non-emergency conditions.

For the CPI, the safety of plant assets and personnel is a crucial concern. Facilities must meet their safety needs while optimizing operational and business performance. Faced with this reality, they are seeking to utilize advanced gas-safety technologies to help ensure the lowest risk and highest level of protection for their most important resources. ■

Edited by Mary Page Bailey

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U.S. Patent Rights: Think Twice Before Disclosing Your Invention

These cautionary tales provide insight that can lead to best practices for scientists who are seeking patents for their inventions

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FIGURE 1. Engineers and scientists who seek patents for their inventions need to understand what precautions to take in order to secure their intellectual property

Publish or perish is a maxim in the scientific community, where scientists are encouraged to share their work quickly and often. Scientists spend considerable time and effort seeking grants, submitting abstracts to conferences and scientific journals, and presenting and publishing their research. They may also collaborate with other scientists, discuss their work with investors, or offer their products for sale. Although each of these activities may lead to prominence or even profits, scientists who act too soon or share too much create a risk of forfeiting their own U.S. patent rights (Figure 1). Cautionary tales of inventors who have risked their own U.S. patent rights provide valuable insight for scientists in developing their own best practices.

Grant applications

Publicly available information in a grant application may present a possible problem for U.S. patent rights. Although most grant applications today are submitted and maintained on a confidential basis, certain information about federally funded grants may be accessible through a request under the Freedom of Information Act (FOIA). If the information includes a description of the claimed invention, and it becomes publicly available more than one year before a U.S. patent application is filed, it may preclude U.S. patent protection for that claimed invention.

For example, in 2014, the U.S. Patent and Trademark Office (USPTO) Patent Trial and Appeal Board (PTAB) considered whether a University of Washington inventor's own grant applications could be used as a basis for finding unpatentable his issued U.S. patent claims related to detecting analytes in liquid media [*1*] The inventor, having obtained grants from the National Human Genome Research Institute, then filed a U.S. patent application more than one year later. The patent issued and several years passed. In 2013, Oxford Nanopore Technologies Ltd. used FOIA requests to obtain redacted copies of the grant applications. Oxford then argued to the PTAB that the patent claims should be found unpatentable because the invention was publicly disclosed in the grant applications more than one year before patent filing, as shown in the 2013 versions of the grant applications. Copies of the grant applications dated more than one year before patent filing were not presented to the USPTO during the original prosecution of the patent or to the PTAB during this later proceeding. The PTAB determined that the 2013 versions of the grant applications did not indicate what was publicly available more than one year before patent filing, because additional information might have been redacted from the public if FOIA requests had been submitted back then. The PTAB thus declined to find the patent claims unpatentable based on prior public disclosure. Although the patent claims were not found unpatentable in this instance, this case may have turned out differently if the PTAB had been presented with publicly available copies of the grant applications dated more than one year before patent filing and they had disclosed the claimed invention.

This cautionary tale demonstrates that scientists should take steps to ensure that their grant applications are submitted and maintained on a confidential basis. In addition, out of an abundance of caution, inventors should consider filing a U.S. patent application within one year of submitting a grant application that describes the invention, or before

submitting a grant application at all if they are considering global patent protection, since not all jurisdictions have the one-year grace period.

Abstracts and papers

Abstracts and papers that are shared or maintained on a non-confidential basis may present a possible problem for U.S. patent rights. As with grant applications, if the abstract or paper describes the claimed invention and becomes publicly available more than one year before a U.S. patent application is filed, U.S. patent protection for that claimed invention may be precluded.

For example, in 1963, the USPTO considered whether an inventor's submission of an advanced proof of a paper to a symposium precluded the inventor from obtaining a U.S. patent relating to distillation of volatile chlorides [*2*]. The inventor submitted an advanced proof of a paper to the Institution of Mining and Metallurgy for discussion at a symposium to be held at the Royal Society of the Arts in London in 1956. The Institution provided copies of the paper to the symposium registrants and attendees, and when the inventor applied for a U.S. patent more than one year later, the examiner rejected the claims over the advanced proof of the symposium paper. The inventor appealed, but the USPTO Board agreed with the examiner. It held that the prior disclosure in the advanced proof of the paper constituted public disclosure of the claimed invention more than one year before patent filing, and U.S. patent protection was thus precluded.

Whether an abstract or paper constitutes a public disclosure is often a fact-intensive inquiry that may turn on the evidence presented. For example, in 2004, the U.S. Court of Appeals for the Federal Circuit considered whether a meeting abstract could be used to invalidate U.S. patent claims owned by Norian Corporation related to tooth and bone repair [*3*]. The inventor had submitted an abstract for a meeting of the International Association for Dental Research (IADR) in 1991. More than one year later, Norian filed a U.S. patent application. The patent issued and Norian subsequently sued Stryker Corporation in the Northern

FIGURE 2. To keep presentations as confidential as possible, audience members may be asked to not take notes or photographs

District of California for infringement. During the litigation, Stryker argued that the patent claims were invalid based on prior disclosure in the IADR abstract. The district court disagreed, finding that the abstract was not disseminated at the IADR meeting and was thus not a prior public disclosure of the claimed invention. In making this finding, the district court relied on one presenter's testimony that he could not recall whether copies of the abstract were available to attendees, and the other presenter's testimony that no one asked him about the availability of the abstract. On appeal, the Federal Circuit affirmed. While Norian's claims were not invalidated in this instance, this case may have turned out differently if the court found, for example, that the presenters or the IADR had distributed copies of the abstract to attendees. Filing a U.S. patent application before submitting the meeting abstract would have avoided this risk to the claims.

As another example, in 2009, the Federal Circuit considered whether a paper that the inventor shared with coworkers could be used to invalidate U.S. patent claims owned by Cordis Corporation related to stents [*4*]. The inventor was a medical resident who prepared a paper in 1980 relating to his work with stents. He provided copies of the paper to about six of his teachers, and orally presented it to them and several other colleagues. The inventor later gave copies of the paper to Vascor, Inc. and Shiley, Inc. while trying to commercialize his stent technology. The agreements between the inventor and the two companies did not require confidentiality, and the Shiley agreement specifically stated that Shiley was not required to keep the information secret. In 1983, the inventor joined the faculty at the University of Texas, San Antonio, and provided copies of a revised version of his paper to a doctor and technician there, and to the university as part of a research proposal. About two years later, the inventor filed a U.S. patent application. A patent issued, and Cordis subsequently sued Boston Scientific Corporation in the District of Delaware for infringement. During the litigation, Boston Scientific responded that the patent claims were invalid based on prior disclosure in the 1980 and 1983 papers. The district court disagreed, and on appeal, the Federal Circuit affirmed. The Federal Circuit held that the papers were not public disclosures of the claimed invention because there were expectations of confidentiality between the inventor and the individuals receiving copies. Although Cordis's claims were not invalidated in this instance, this case may have turned out differently if the court had determined, for example, that some of the recipients were not expected to keep the paper confidential. This risk to the claims could have been avoided by filing a U.S. patent appli-

cation before the 1980 papers were disseminated.

A publicly available abstract or paper that discloses the claimed invention more than one year before patent filing may present a possible problem for U.S. patent rights, even if the authors do not realize the significance of their work at the time they write the abstract. For example, in 2006, the Federal Circuit considered whether an abstract that the inventors published in the *European Journal of Pharmaceutical Scientists* could be used to invalidate U.S. patent claims owned by Nichols Institute Diagnostics, Inc. relating to antibodies for human parathyroid hormone [*5*]. The inventors published the abstract in 1994, and later realized the significance of their work. One year and ten days after the abstract was published, the inventors filed a U.S. patent application. A patent issued, and Nichols Institute Diagnostics subsequently sued Scantibodies Clinical Laboratory, Inc. in the Southern District of California for infringement. During the litigation, Scantibodies argued that the patent claims were invalid based on prior disclosure in the published abstract. The district court disagreed, finding that the abstract did not constitute a public disclosure of the claimed invention because the inventors did not know the significance of their work when the abstract was published. On appeal, however, the Federal Circuit overturned the district court decision, holding that the patent claims were invalid, because the abstract disclosed the claimed invention to the public more than one year before the U.S. patent application was filed and the significance of the work was inherent in the disclosure.

Learning from these cautionary tales, scientists should take steps to ensure that abstracts and papers describing their inventions are submitted to conferences and journals or shared with colleagues on a confidential basis. As an even better course, if practicable, inventors should consider filing a U.S. patent application within one year of submitting or sharing their abstract or paper, or, better yet, before submitting or sharing their abstract or paper at all if they are considering global patent protection.

Presentations

Presentations that constitute public disclosure of a claimed invention more than one year before patent filing may present a possible problem for U.S. patent rights. For example, in 1985, the Federal Circuit considered whether a presentation by the inventors could be used to invalidate U.S. patent claims owned by the Massachusetts Institute of Technology (MIT) related to biological cell cultures [*6*]. The inventors had presented their work on microcarriers at the First International Cell Culture Congress in September 1976. The conference was attended by fifty to five-hundred cell culturists, and printed copies of the presentation were distributed to a number of scientists. One year and two months after the conference, the inventors filed a U.S. patent application. A patent issued, and MIT asserted it against AB Fortia at the International Trade Commission (ITC). The ITC determined that the patent claims were invalid because the presentation and handouts constituted public disclosure of the claimed invention more than one

year before patent filing. On appeal, the Federal Circuit agreed.

As with abstracts and papers, whether a presentation constitutes a prior public disclosure of the claimed invention is often a fact-intensive inquiry. It may turn on who had access to the presentation, whether the access was confidential, and what the audience could learn about the claimed invention from the presentation. For example, in 1981, the U.S. District Court for the District of New Jersey considered whether a presentation by the inventors could be used to invalidate U.S. patent claims owned by the Regents of the University of California related to prosthetic knees [*7*]. The inventors had presented lectures about their work to approximately thirty physicians at the 101st Annual Session of the California Medical Association in 1972. One year and two days later, the inventors filed a U.S. patent application. The patent issued, and the Regents of the University of California subsequently sued Howmedica, Inc. in the District of New Jersey for infringement. During the litigation, Howmedica argued that the patent claims were invalid based on prior disclosure during the 1972 lectures. The district court disagreed, reasoning that even though the audience was not asked to keep the information confidential, the slides were projected only temporarily and did not adequately disclose how to make and use the claimed invention. Although the Regent's claims were not invalidated in this instance, this case may have turned out differently if the court had found, for example, that the slides had been projected for additional time or that they provided more information about the invention. This risk to the patent claims could have been avoided if a U.S. patent application had been filed before the presentation.

As another example, in 2004, the Federal Circuit considered whether a presentation by the inventors at two different meetings precluded them from obtaining U.S. patent protection [*8*]. The inventors had presented their research relating to cholesterol at a meeting of the American Association of Cereal Chemists (AACC) and at an Agriculture Experiment Station (AES) at Kansas State University in 1998. The presentations involved displaying a poster with fourteen printed slides for about three days total. About two years later, the inventors filed a U.S. patent application. The USPTO examiner rejected the claims based on prior disclosure of the claimed invention at the two meetings. The inventors appealed to the USPTO Board, which agreed with the examiner, and then to the Federal Circuit, which also agreed with the examiner. The Federal Circuit reasoned that the presentation was a prior public disclosure of the claimed invention because the inventors displayed their poster for about three days to a target audience of cereal chemists, the audience was not precluded or discouraged from taking notes or photographs, and only a few slides on the poster needed to be copied for the claimed invention to be captured.

FIGURE 3. **Scientists should avoid demonstrations of and communications about their inventions, especially in informal settings**

A presentation does not need to involve the inventor to present a possible problem for U.S. patent rights. For example, in 2016, the USPTO considered whether a presentation by an undergraduate student could be used to invalidate U.S. patent claims owned by the University of Washington relating to detecting analytes in liquid media [*9*]. The student had presented work from the laboratory at a 2007 University of Washington undergraduate poster session. Several years later, the University of Washington filed a U.S. patent application related to the work. The patent issued, but Oxford Nanopore Technologies Ltd. subsequently petitioned the USPTO to find the issued patent claims unpatentable based on prior disclosure in the undergraduate poster presentation. The USPTO Board disagreed, concluding that the presentation was not a public disclosure of the claimed invention because the poster session included a broad range of topics and that the viewers were primarily affiliated with the students. Although the University of Washington's claims were not found unpatentable in this instance, this case may have turned out differently if the Board had determined, for example, that the posters were all related to the subject matter of the invention or if the audience included more scientists in the field. This risk to the claims could have been avoided by filing a U.S. patent application before the presentation.

Learning from these cautionary tales, scientists should keep their presentations as confidential as possible, for example, by asking that audience members not take notes or photographs and by displaying slides only temporarily, especially if the target audience includes scientists in the same field as the invention (Figure 2). In addition, out of an abundance of caution, inventors should consider filing a U.S. patent application within one year of presenting their work, or before presenting their work at all if they are considering global patent protection.

Use

Public use of a claimed invention more than one year before patent filing may present a possible problem for U.S. patent rights. For example, in 2005, the Federal Circuit considered whether use of the invention in Invitrogen Corporation laboratories invalidated its U.S. patent claims [*10*]. Invitrogen had used its inventive process for producing competent *E. coli* cells in its laboratories for more than one year before filing a U.S. patent application. A patent issued, and Invitrogen subsequently sued Stratagene Holding Corporation in the Western District of Texas for infringement. During the litigation, Stratagene argued that the patent claims were invalid based on prior use. The district court agreed. On appeal, however, the Federal Circuit disagreed and determined that Invitrogen's use did not invalidate its patent claims because it was not a public use of the claimed invention. The court reasoned that Invitrogen did not sell the claimed process or any products made using the process, and it maintained the process as a secret within the company. Although Invitrogen's claims were not invalidated in this instance, this case may have turned out differently if the court found, for example, that Invitrogen's use of the invention was not kept secret.

This cautionary tale demonstrates that scientists should keep any use of their inventions confidential. In addition, out of an abundance of caution, inventors should consider filing a U.S. patent application within one year of using their invention, or before using their invention at all if they are considering global patent protection.

Demonstrations and communications

Demonstrations of a claimed invention and communications about a claimed invention more than one year before patent filing may present a possible problem for U.S. patent rights. For example, in 1972, the U.S. District Court for the Northern District of Illinois considered whether an inventor's demonstration of the invention and a subsequent discussion at an inter-company meeting could be used to invalidate U.S. patent claims [*11*]. Seeking to sell his invention, the inventor had demonstrated his machine for laying cable and other materials underground to an engineer at the Illinois Bell Telephone Company in 1963. The Bell Telephone engineer provided a written report to his supervisor, who then read it during an inter-company meeting of telephone company engineers. One year and three months after the meeting, the inventor filed a U.S. patent application. A patent issued, and the owner subsequently sued J.I. Case Co. in the Northern District of Illinois for infringement. During the litigation, J.I. Case argued that the patent claims were invalid based on prior disclosure during the 1963 demonstration and inter-company meeting. The district court agreed, reasoning that the demonstration and meeting were public

disclosures of the claimed invention because there was no confidential relationship between the inventor and the engineer or the company.

A demonstration or communication need not involve the inventor to present a possible problem for U.S. patent rights. For example, in 1994, the Federal Circuit considered whether a conversation involving the inventor's graduate advisor could be used to invalidate U.S. patent claims owned by National Research Development Corporation (NRDC) [*12*]. The graduate advisor had attended an Experimental Nuclear Magnetic Resonance (NMR) Conference in the United States in 1973. On the bus to the conference, he had an informal and non-confidential conversation with a scientist from Monsanto Company about the inventor's work on NMR. Monsanto subsequently incorporated the invention into an NMR spectrometer it used for analyzing herbicides. About two years later, the inventor filed a U.S. patent application. A patent issued, and NRDC subsequently sued Varian Associates, Inc. in the District of New Jersey for infringement. During the litigation, Varian argued that the patent claims were invalid based on the 1973 disclosure by the graduate advisor and Monsanto's subsequent use. The district court agreed, and on appeal, the Federal Circuit affirmed. The Federal Circuit reasoned that the graduate advisor's unrestricted disclosure of the claimed invention more than one year before patent filing, coupled with the use of that information in the ordinary course of Monsanto's business, invalidated the patent claims.

Learning from these cautionary tales, scientists should ensure that any demonstrations of their inventions and communications about their inventions are kept confidential (Figure 3). In addition, out of an abundance of caution, inventors should consider filing a U.S. patent application within one year of demonstrating or communicating about their invention, or before demonstrating or communicating about their invention at all if they are considering global patent protection.

Sales and offers to sell

Sales of a claimed invention and offers to sell a claimed invention more than one year before patent filing may present a possible problem for U.S. patent rights. For example, in 2016, the Federal Circuit considered whether Ben Venue Laboratories' supply of batches of an anticoagulant product could be used to invalidate patent claims owned by the Medicines Company (MedCo) relating to a process for making the product [*13*]. More than one year after MedCo contracted with Ben Venue to manufacture three batches of MedCo's anticoagulant product, MedCo filed a U.S. patent application. A patent issued, and MedCo subsequently sued Hospira Inc. in the District of Delaware for infringement. During the litigation, Hospira argued that the patent claims were invalid based on Ben Venue's sale of the product to

MedCo more than one year before patent filing. The district court disagreed, determining that the transaction between MedCo and Ben Venue was not a commercial offer for sale of the claimed invention because MedCo retained title to the product and the three batches were supplied for experimental purposes rather than commercial profit. On appeal, the Federal Circuit affirmed. While Ben Venue's claims were not invalidated in this instance, this case may have turned out differently if the court had determined, for example, that the batches were supplied for commercial profit. This risk to the claims could have been avoided by filing a U.S. patent application before batches of the product were supplied.

A sale does not need to actually occur for an offer of sale more than one year before patent filing to present a possible problem for U.S. patent rights. For example, in 2016, the Federal Circuit considered whether a fax with price, delivery, and payment terms could be used to invalidate U.S. patent claims owned by Merck & Cie even though a sale did not take place [*14*]. A Merck manager had sent a fax in 1998 to Weider Nutrition International relating to the sale of a crystalline calcium salt of tetrahydrofolic acid. About one and half years later, Merck filed a patent application. A patent issued, and Merck sued Watson Laboratories, Inc. in the District of Delaware for infringement. During litigation, Watson Laboratories argued that the patent claims were invalid based on an offer for sale more than one year before patent filing. The district court disagreed, finding that there was no legally binding sale of the claimed invention because the agreement had not been reduced to writing and signed by both parties. On appeal, the Federal Circuit reversed, holding that the fax was an offer for sale that could invalidate the patent claims even though a sale never occurred, because both parties understood it to be an offer for sale of the claimed invention.

A sale more than one year before patent filing only presents a possible problem if the invention is ready for patenting at the time of the sale. Whether an invention is ready for patenting depends on the facts of the case and does not necessarily require a working prototype. For example, in 1988, the U.S. Supreme Court considered whether an inventor's commercial offer for sale could be used to invalidate U.S. patent claims related to computer chip sockets [*15*]. The inventor had offered to sell his computer chip socket in commercial quantities to Texas Instruments in 1981. Although he had not yet made a working prototype, he showed sketches of the design concepts to Texas Instruments in relation to the offer for sale. One year and eleven days after confirmation by Texas Instruments, the inventor filed a U.S. patent application. A patent issued, and the inventor subsequently sued Wells Electronics, Inc. in the Northern District of Texas for infringement. During litigation, Wells Electronics argued that the patent claims were invalid based on the offer for sale more than one year before patent filing. The district court decided that some of the patent claims were invalid. On appeal, the Federal Circuit reversed, holding that all the patent claims were invalid based on an offer for sale of the claimed invention more than one year before patent filing. It reasoned that even though the inventor did not have a working prototype at the time he offered his product for sale, the invention was substantially complete. On appeal, the U.S. Supreme Court affirmed.

Learning from these cautionary tales, scientists should consider filing a U.S. patent application within one year of offering their invention for sale, or before offering it for sale at all if they are considering global patent protection.

Closing thoughts

Placing an invention into the public domain more than one year before filing a U.S. patent application may present a possible problem for U.S. patent rights. Scientists should thus think twice before disclosing their inventions when seeking grants, submitting abstracts, presenting, publishing, and communicating with others, particularly if they have not yet filed a U.S. patent application. As reflected in cautionary tales of other inventors, the circumstances and timing of the disclosure may determine whether a scientist inadvertently forfeits their U.S. patent rights. ■

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Economic Indicators

2016 ▬ **2017** ▬ **2018** ▬ **2019** ▬

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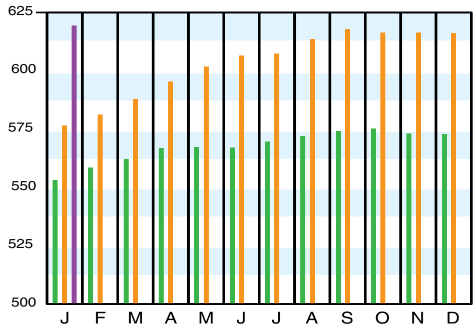
CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957–59 = 100)	Jan. '19 Prelim.	Dec. '18 Final	Jan. '18 Final
CE Index	619.2	615.9	576.4
Equipment	757.5	751.2	697.4
Heat exchangers & tanks	677.3	667.3	606.1
Process machinery	734.3	731.2	697.0
Pipe, valves & fittings	978.9	979.9	910.2
Process instruments	416.1	420.2	415.9
Pumps & compressors	1060.6	1037.3	1001.0
Electrical equipment	554.7	553.7	531.2
Structural supports & misc.	841.7	827.2	736.1
Construction labor	334.1	339.5	328.7
Buildings	601.5	600.1	570.5
Engineering & supervision	316.9	316.3	308.7

Starting in April 2007, several data series for labor and compressors were converted to accommodate series IDs discontinued by the U.S. Bureau of Labor Statistics (BLS). Starting in March 2018, the data series for chemical industry special machinery was replaced because the series was discontinued by BLS (see *Chem. Eng.*, April 2018, p. 76–77.)

Annual Index:

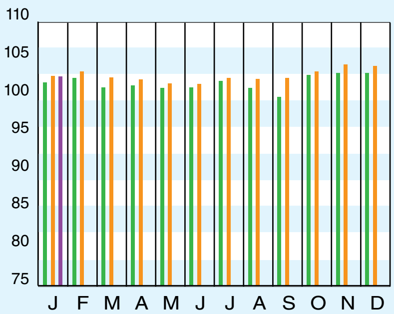
2011 = 585.7
2012 = 584.6
2013 = 567.3
2014 = 576.1
2015 = 556.8
2016 = 541.7
2017 = 567.5
2018 = 603.1



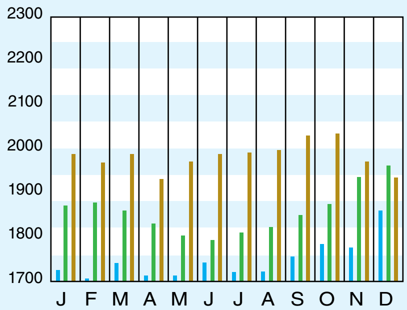
CURRENT BUSINESS INDICATORS

	LATEST	PREVIOUS		YEAR AGO
CPI output index (2012 = 100)	Feb. '19 = 102.7	Jan. '19 = 103.6	Dec. '18 = 104.1	Feb. '18 = 101.9
CPI value of output, $ billions	Jan. '19 = 1,935.9	Nov. '18 = 1,943.4	Oct. '18 = 1,971.7	Jan. '18 = 1,937.9
CPI operating rate, %	Feb. '19 = 76.1	Jan. '19 = 76.7	Dec. '18 = 77.2	Feb. '18 = 76.1
Producer prices, industrial chemicals (1982 = 100)	Feb. '19 = 255.1	Jan. '19 = 249.7	Dec. '18 = 260.7	Feb. '18 = 270.3
Industrial Production in Manufacturing (2012 =100)*	Feb. '19 = 104.8	Jan. '19 = 105.2	Dec. '18 = 105.7	Feb. '18 = 103.8
Hourly earnings index, chemical & allied products (1992 = 100)	Feb. '19 = 185.2	Jan. '19 = 187.8	Dec. '18 = 186.9	Feb. '18 = 187.9
Productivity index, chemicals & allied products (1992 = 100)	Feb. '19 = 94.1	Jan. '19 = 95.5	Dec. '18 = 96.9	Feb. '18 = 95.8

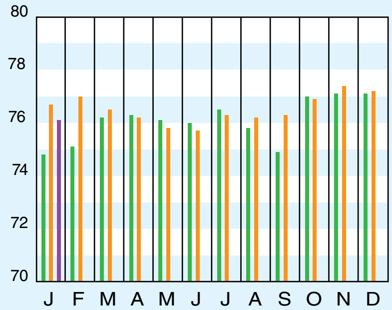
CPI OUTPUT INDEX (2000 = 100)†



CPI OUTPUT VALUE ($ BILLIONS)



CPI OPERATING RATE (%)



*Due to discontinuance, the Index of Industrial Activity has been replaced by the Industrial Production in Manufacturing index from the U.S. Federal Reserve Board.
†For the current month's CPI output index values, the base year was changed from 2000 to 2012
Current business indicators provided by Global Insight, Inc., Lexington, Mass.

CURRENT TRENDS

Since the final value for the December 2018 *CE* Plant Cost Index (CEPCI; top) is now available, it is possible to calculate the 2018 CEPCI annual average value, which is 603.1. The total represents a 6.3% rise over the annual value from the previous year. The first preliminary value for 2019 also shows an increase over the final December value, where gains in the Equipment, Engineering & Supervision, and Building subindexes offset a decrease in the Construction Labor subindex. The overall CEPCI preliminary value for January 2019 stands at 7.4% higher than the corresponding value from January 2018. Meanwhile, the CBI numbers (middle) show a small decrease in the CPI output index for February 2019.